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TESTING FRAME 4 OF THE MULTI-PURPOSE PROCESSING FACILITY

OCTOBER 1979

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**TESTING FRAME 4 OF THE
MULTI-PURPOSE PROCESSING FACILITY**

OCTOBER 1979

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CONTENTS

	PAGE
SUMMARY	7
BACKGROUND	8
TEST PROCEDURE	8
TEST RESULTS	14
Tests with Water	14
Transfer Rates	15
Sump Pan Flush Test and Calibration	15
Cooling Water Flow Rates	15
Heating Water Flow Rates	15
Equipment Tests	18
Evaporator Liquid-Level Calibration	18
Samplers	18
Vessel Off-Gas Exhaust System	18
Minimum Volume Heater Operation	23
Heat Transfer to the Evaporator Cooling Coils	24
Heat Transfer to the Evaporator Condenser	24
Heat Transfer from Feed Tank	25
Pressure Drops Across Empty Ion-Exchange Columns and Piping	25
Pressure Drops Across Empty Ion-Exchange Columns Only	25
Pressure Drop Through Piping Only	30
Pressure Drops Across Resin-Loaded Ion-Exchange Columns	30
Temperature in Resin-Loaded Ion-Exchange Columns	30
Removal of Resin from Dump Tank.	37
Cold Chemical Runs	38
Evaporation, Steam-Stripping, and Denitration	38
Accuracy of the Evaporator Specific-Gravity Bubbler	39
Product Hold-up in the Feed Evaporator and Filter	43
RIX-I Displacement Chromatography	43
SYSTEM FLUSH	53
APPENDICES	55
A. Equipment List	55
B. Water Transfer Tests	56
C. Measurement of Vessel Off-Gas Exhaust Flows	57
D. Evaporation, Steam Stripping, and Denitration	63
E. Feed Compositions for RIX-I Chemical Runs	71
F. Displacement Chromatography Runs 1 and 2	73

LIST OF TABLES

	PAGE
1. Cooling Water Flow Rates.	17
2. Heating Water Flow Rates.	17
3. Test of Volume Calibration of Evaporator.	19
4. Sampler Fill and Recirculation Rates.	21
5. Sampler Air Bleed Orifice Size	21
6. Off-Gas Exhaust Flow Requirements for Frame 4 Vessels	22
7. Minimum Amount of Liquid for Operation of Heaters in RIX-I Feed Evaporator	23
8. Measured Flow Rates and Pressure Drops for Empty Ion-Exchange Columns and Associated Piping . . .	26
9. Calculated Maximum Pressure Drop for Empty Columns With Two Frits in Series	29
10. Measured Pressure Drop Compared to Calculated Maximum Pressure Drop for Columns With One Frit and No Resin	31
11. Pressure Drop Across Empty One-Inch Column After Replacement of Top Frit	32
12. Measured Flow Rates and Pressure Drops for Piping and Valves Only	33
13. Pressure Drops Across Resin-Loaded RIX-I Columns in Loading Station	34
14. Pressure Drops Across Resin-Loaded RIX-I Columns in Frame 4	35
15. Temperature of Feed Water Entering and Leaving Resin-Loaded Columns During Heating Tests	36
16. Removal of Resin From Dump Tank	37
17. Composition of Evaporator Bottoms and Over-heads During Evaporation and Steam Stripping	40
18. Entrainment of Evaporator Bottoms in Condensate	41
19. Total H^+ Concentration During Denitration With Formic Acid	41

LIST OF TABLES (Cont'd)

	PAGE
20. Total NO_3^- Concentration During Denitration With Formic Acid	41
21. Verification of Accuracy of Evaporator Specific-Gravity Bubbler	42
22. Feed Evaporator and Filter Transfer Heel for Run 1 . .	44
23. Feed Evaporator and Filter Transfer Heel for Run 2 . .	45
24. Feed Evaporator and Filter Transfer Heel for Run 3 . .	46
25. RIX-I Column Operating Pressures During Nonradioactive Chemical Runs	50
26. RIX-I Column Temperatures During Nonradioactive Chemical Runs	50
27. Percent of Feed Left in Feed Tank After Water Displacement to RIX-I Columns	52
28. Volumes of Dowex® 50W-X8 Resin in RIX-I Columns . . .	52
A-1 Equipment List	55
B-1 Water Transfer Tests of Frame 4 Vessels	56
C-1 Off-Gas Exhaust Flows from Frame 4 Tanks	58
D-1 Run 1 Evaporation, Steam Stripping, and Denitration . .	63
D-2 Run 2 Evaporation, Steam Stripping, and Denitration . .	64
D-3 Run 3 Evaporation, Steam Stripping, and Denitration . .	65
D-4 Run 4A Evaporation, Steam Stripping, and Denitration .	66
D-5 Run 4B Second Denitration	68
D-6 Run 5 Evaporation, Steam Stripping, and Denitration . .	69
D-7 Run 6 Denitration Without Evaporation or Steam Stripping	70
E-1 Feed Make-up for RIX-I Column Runs 1 and 2	71
E-2 Analysis of Feed Solutions for RIX-I Column Runs Runs 1 and 2	72
F-1 Analysis of Effluent From RIX-I Columns During Run 1	73
F-2 Analysis of Effluent From RIX-I Columns During Run 2	77

LIST OF FIGURES

	PAGE
1. Frame 4 Flow Diagram	9
2. Half-Scale Model of Frame 4	10
3. Front-Right View of Frame 4	11
4. Front-Left View of Frame 4	12
5. Calibration of Sump and Pan	16
6. Measurement of Sample Recirculation Rate	20
7. Measurement of Sample Fill Rate	20
8. Off-Gas Exhaust System for Frame 4 Vessels	22
9. Elution Profiles for Run 1 with Nonradioactive Lanthanides in RIX-I Columns	48
10. Elution Profiles for Run 2 with Nonradioactive Lanthanides in RIX-I Columns	49
C-1. Off-Gas Exhaust Flow from Condensate Tank (EP 10-1-10)	59
C-2. Off-Gas Exhaust Flow from RIX-I Dump Tank (EP 10-3-3)	60
C-3. Off-Gas Exhaust Flow from RIX-I Waste Tank (EP 10-3-20)	61
C-4. Off-Gas Exhaust Flow from Cf RIX Run Tank (EP 10-9-1)	62

SUMMARY

Semiworks tests of the Multi-Purpose Processing Facility (MPPF) Frame 4 were completed. The part of the Californium-I process performed in this frame includes: (1) feed adjustment by evaporation, steam stripping, and formic acid denitration, and (2) separation of californium, berkelium, curium, and americium from the lanthanide fission products by displacement-development chromatography using rapid ion exchange (RIX-I). This report describes these tests and the modifications required to attain intended frame performance. Highlights of the test results, some of which necessitated minor design changes, are summarized below.

- Transfer rates were satisfactory through all process paths after minor equipment modifications. The top frit was replaced in the 1-inch-diameter RIX-I column (EP 10-3-13) to eliminate excessive pressure drop which resulted during flow testing of the empty (without resin bed) column.
- The off-gas exhaust (OGE) vent valve #465 in transfer line #505 between the dissolved cake tank (EP 10-19-9) and the evaporator transfer tank (EP 10-1-5) was found to be incorrectly installed downstream from line valve #464, instead of upstream as shown on the piping diagram. This was corrected so that line #505 can be vented while the evaporator transfer tank is evacuated.
- Vent valve #444 in transfer line #133 between the RIX-I feed tank (EP 10-3-1) and the evaporator transfer tank (EP 10-1-5) was found to siphon the contents of the feed tank into the dump tank (EP 10-3-3). The siphon was eliminated by replacing valve #444 with a rupture disc.
- The spare dipleg line #SP 201 from the evaporator transfer tank (EP 10-1-5) was found to siphon the contents from the transfer tank when the cap was removed from the nozzle on the panel board. The siphon was eliminated by evacuating the transfer tank before removing the cap from the nozzle.
- Calibration of the liquid-level float mechanism for the evaporator transfer tank (EP 10-1-5) was not reproducible when the transfer tank was evacuated. Air leakage at the connection unions was shown to affect the liquid level reading. The problem, which was not eliminated by tightening the unions, has not yet been resolved satisfactorily.

- Sampler fill and recirculation rates were satisfactory.●
- Off-gas flow rates at a vacuum of 0.1 inch of water met or exceeded design requirements for diluting radiolytic hydrogen in all vessels.
- Chemical runs simulating actual flowsheet conditions indicated satisfactory equipment performance and run times.

BACKGROUND

The part of the Californium-I process that is performed in Frame 4 is shown in Figure 1. For feed adjustment, about 50 liters of Cf-I solution are transferred from the 17.3E canyon evaporator to the MPPF feed evaporator (EP 10-1-8E) for each run, along with 20 liters of feed tank backcycle solution and about 3 liters of Cm-Am-Eu overlap fraction from the recycle conversion step. Feed preparation is based on a batch containing about 150 mg of ^{252}Cf and about 2.2 moles of actinides plus lanthanides. The feed batch is evaporated to about 10 liters, steam stripped to about 4M HNO_3 , denitrated to about 1M HNO_3 with formic acid, treated with NaNO_2 , and then diluted to 20 liters in the RIX-I feed tank. The feed is passed through the 4-inch-diameter RIX-I column by displacement from the feed tank with 60 liters of deionized water. The RIX-I columns contain Dowex® 50W-X8 (Dow Chemical Co.) cation-exchange resin previously saturated with Zn^{2+} barrier ion before each run. The californium, berkelium, curium, and americium are separated from the lanthanide fission products by displacement development chromatography with 0.05M diethylenetriaminepentaacetic acid (DTPA) buffered to pH 6.0 with NH_4OH .

Figure 2 is a photograph of the half-scale model from which the frame was constructed. Figures 3 and 4 are front-right and front-left views of the completed frame. EP numbers and capacities of the equipment on Frame 4 are listed in Appendix A, which also lists references to detailed drawings of the equipment and process arrangement.

TEST PROCEDURES

The test program comprised (1) continuity, operability, and hydraulic checks with water, (2) specific equipment testing, and (3) cold chemical runs with synthetic, nonradioactive process solution.

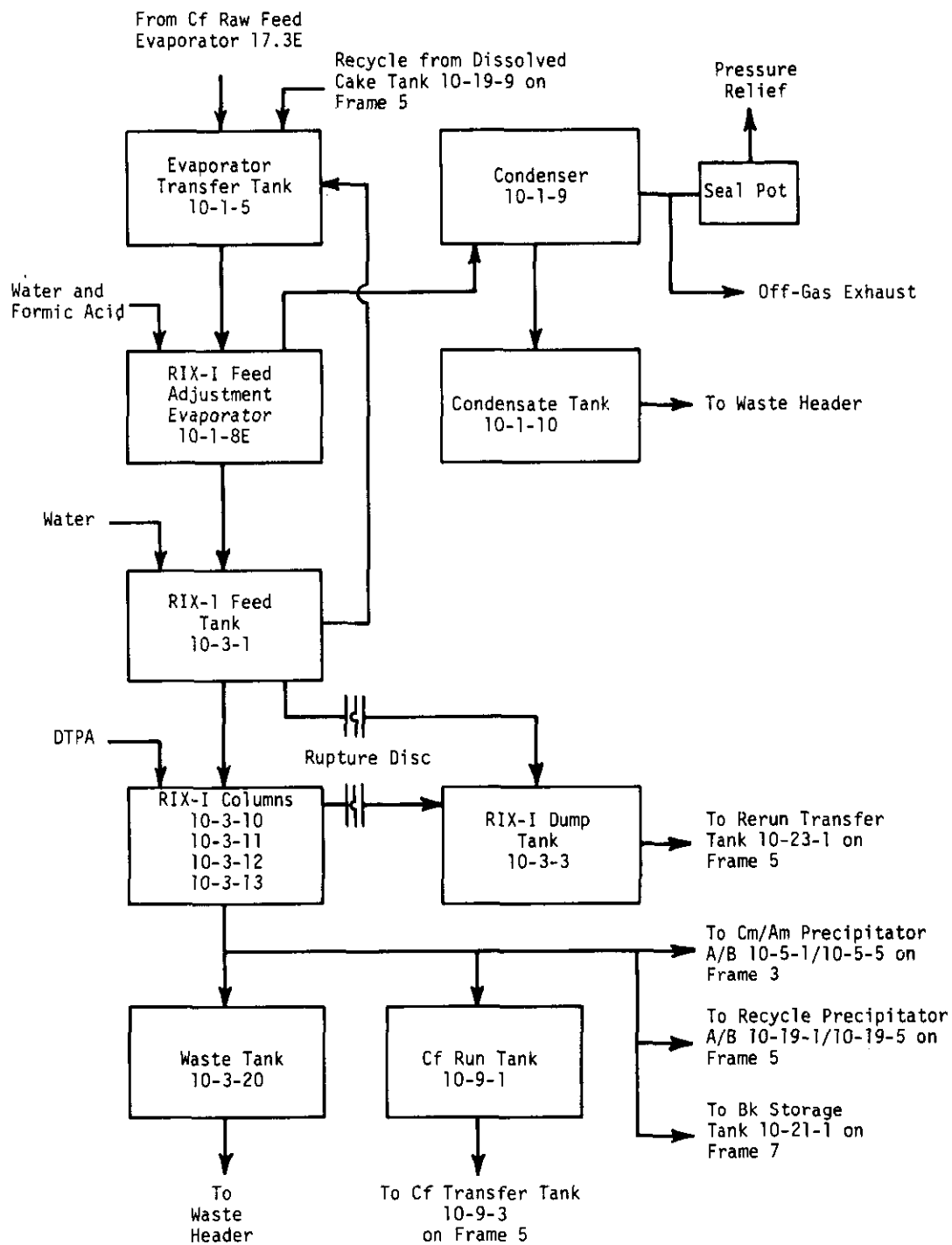


FIGURE 1. Frame 4 Schematic Flow Diagram

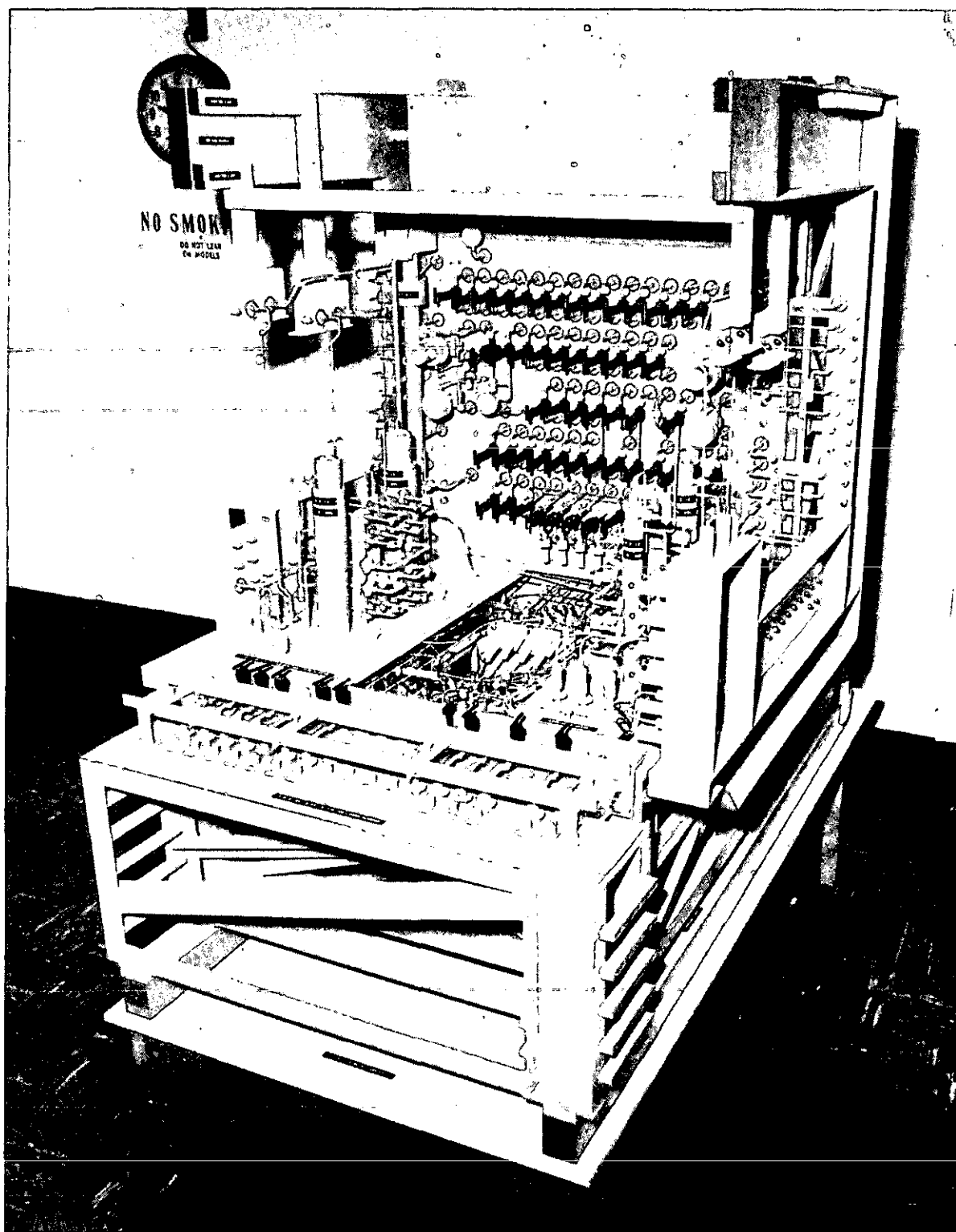


FIGURE 2. Half-Scale Model of Frame 4

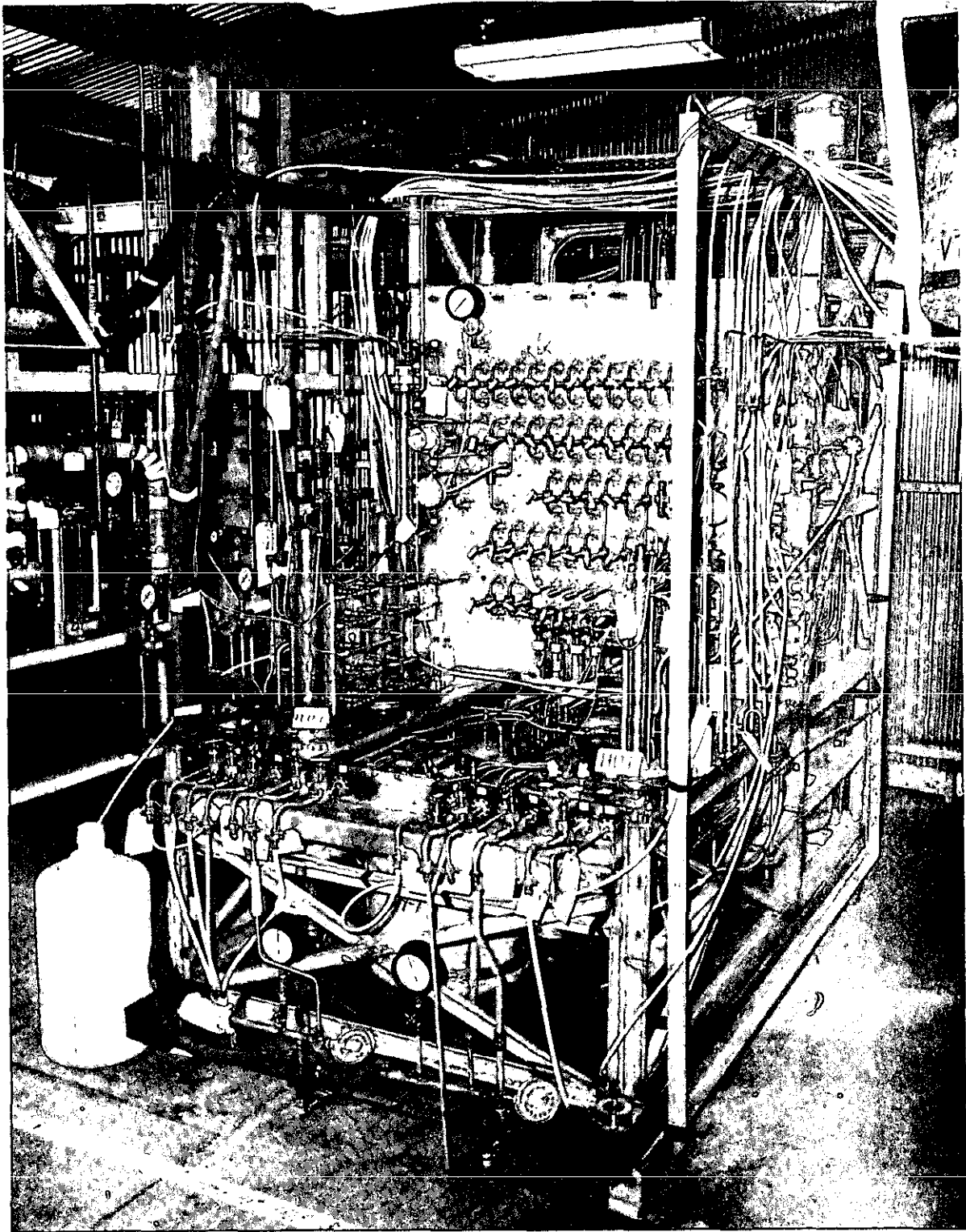


FIGURE 3. Front-Right View of Frame 4

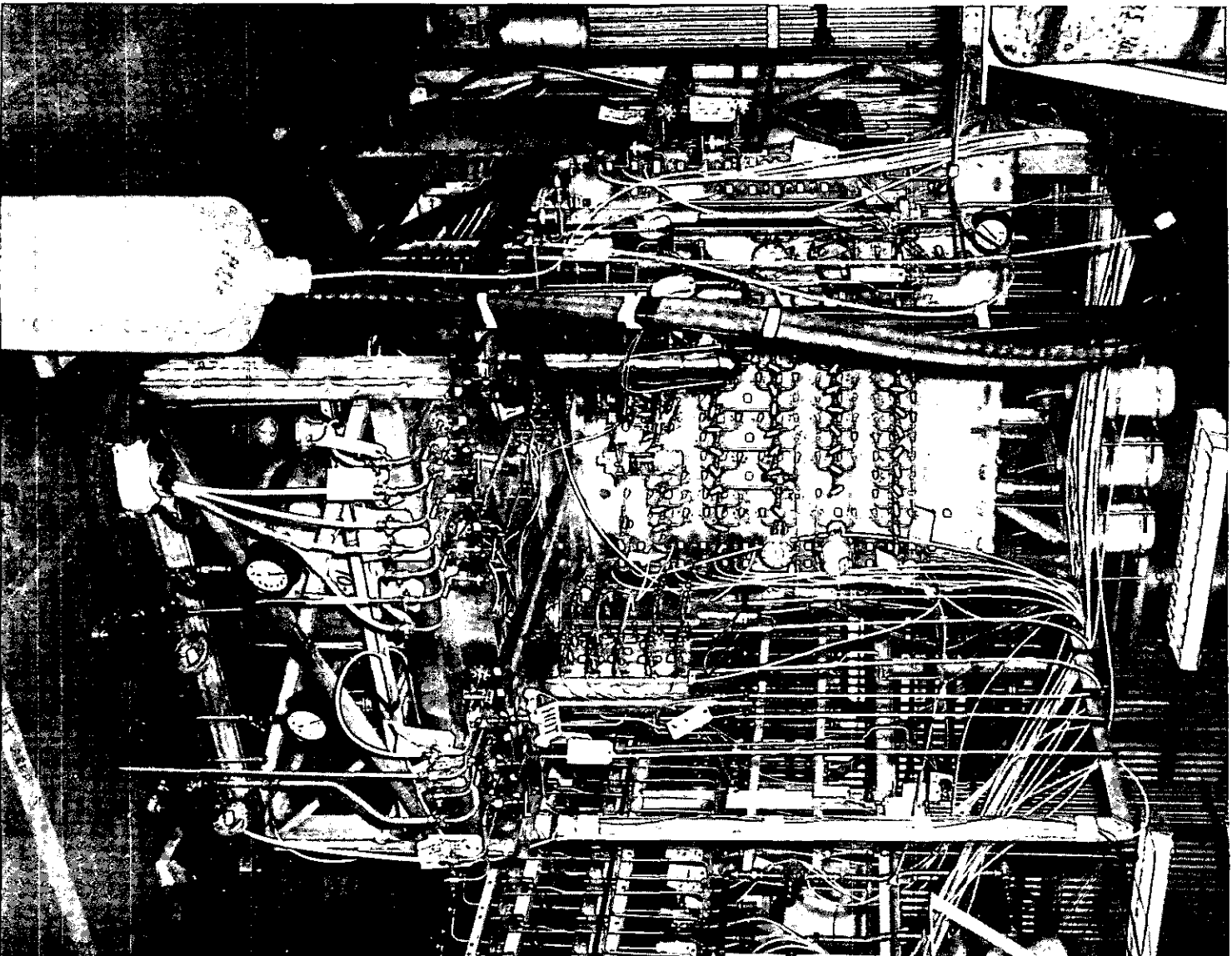


FIGURE 4. Front-Left View of Frame 4

TESTS WITH WATER

Continuity, operability, and hydraulic characteristics of each process vessel and line were tested with filtered, deionized water. Specific items investigated are listed below:

- Lines into Vessels: all lines into vessels, with the exception of the off-gas and evacuation jet lines, were tested by flowing water into the vessel by pump, gravity, or vacuum transfer.
- Thermowells: satisfactory operation of thermocouples in all vessels containing thermowells was verified by heating water in each vessel.
- Samplers: each sampler was operated to verify that its recirculation rate was acceptable.
- Vacuum Transfer: all vacuum transfer systems were tested to ensure adequate evacuation capacity.
- Cooling and Heating Water: flow of cooling and heating water to equipment with jackets or coils was measured.
- High Pressure Lines: all lines and equipment that operate under high pressure were tested at pressures up to 1000 psig to ensure adequate flow capacity.
- Evaporator: the evaporator was tested to determine its boilup rate and to verify that the condenser will condense the off-gas vapors. The boilup rate and the minimum volume required for heater operation were determined.
- Rod-Out Ports: sufficient internal clearance of all rod-out ports was verified.
- Sump: the frame sump was cleaned and calibrated. The spray-down line was operated to determine distribution coverage of the sump pan.

EQUIPMENT TESTS

Specific pieces of equipment were tested in detail, including extensive measurements of the venting capacity of each process vessel.

COLD CHEMICAL RUNS

Three full-scale evaporation, steam stripping, and denitration runs were made with nitric acid and formic acid. Three more full-scale evaporation, steam stripping, and denitration runs were made with nitric acid, formic acid, and added neodymium nitrate as a stand-in for the actinide-lanthanide mixture present in a radioactive feed.

Two full-scale chemical runs were made through the RIX-I columns using dysprosium, terbium, gadolinium, europium, samarium, and neodymium as stand-ins for the actinide-lanthanide mixture present in radioactive feed.

TEST RESULTS

Tests with Water

Transfer Rates. Data from these tests are listed in Appendix B. Cold feed drops were made by gravity via 3/8-inch-diameter polyethylene tubing from a bottle about 6 feet above the rack deck. Measurements of vacuum transfer rates were started after the transfer tank vacuum exceeded 20 inches of mercury. Positive displacement pumps provided water flow through high pressure lines and equipment. Transfer rates through all process paths were satisfactory after minor modifications. The top frit of the 1-inch-diameter RIX-I column (EP 10-3-13) was replaced to eliminate the excessive pressure drop during flow testing of the empty (without resin bed) column. The off-gas exhaust vent valve #465 in transfer line #505 between the dissolved cake tank (EP 10-19-9) and the evaporator transfer tank (EP 10-1-5) was found to be incorrectly installed downstream from transfer valve #464, instead of upstream as shown on the piping diagram. The purpose of vent valve #465 is to break the vacuum and ensure that solution would not be transferred if the transfer valve #464 leaked. With the incorrect installation, the evaporator transfer tank could not be evacuated with the vent valve open and the transfer line could not be vented with the transfer valve closed. The installation was corrected so that line #505 can be vented while the evaporator transfer tank is evacuated.

Vent valve #444 in transfer line #133 between the RIX-I feed tank (EP 10-3-1) and the evaporator transfer tank (EP 10-1-5) was found to siphon the contents of the feed tank into the dump tank (EP 10-3-3). Vent valve #444 is located on the panel board below the elevation of the feed tank. Evacuation of the feed tank filled line #133 with liquid up to vent valve #444. Line #133 remained full of liquid after the feed tank vacuum was relieved. Opening valve #444 siphoned the contents of the feed tank into the

dump tank. The siphon was eliminated by replacing valve #444 with a rupture disc.

The nozzle for the spare dip line #SP 201 from the evaporator transfer tank (EP 10-1-5) was found to siphon the contents from the transfer tank when the cap was removed from the nozzle. The nozzle is located on the panel board below the elevation of the transfer tank. Evacuation of the transfer tank filled the line with liquid up to the capped nozzle. The line remained full of liquid after the vacuum was relieved in the transfer tank. Removal of the cap from the nozzle siphoned the contents of the transfer tank into the sump. The siphon was eliminated by evacuating the transfer tank before removing the cap from the nozzle.

Sump Pan Flush Test and Calibration. The effectiveness of the Frame 4 pan flush system was tested with a water flow of 2.45 liters/minute at a supply pressure of 10 psig. Distribution of flush water over the pan area was very poor; there were four small separate streams flowing onto the pan instead of sprays distributed uniformly over the entire pan. The waviness of the pan created a low spot under the condensate tank (EP 10-1-10) about 1/8-inch deep and 3 feet in radius that collected liquid which would not drain into the sump. The sump was filled to the level of the pan with 5.39 liters of water at a sump liquid-level reading of 2.95 inches. The pan was straightened and six more holes were added to improve distribution of the flush over the pan area.

The sump and pan surfaces were thoroughly cleaned. The sump and pan volume were calibrated by adding measured amounts of water and reading the liquid level manometer after each addition. The calibration is shown in Figure 5.

Cooling Water Flow Rates. Cooling water flow rates to vessels with jackets or coils were measured (Table 1). Flow rates were highest when only one vessel jacket or coil was valved on at a time. Flow rates decreased as each additional vessel jacket or coil was valved on. The decrease in flow is attributed to increased pressure drop for water flow through the common supply header.

Heating Water Flow Rate. Heating water flows through the jackets of the four RIX-I columns in series, entering the bottom of the 4-inch diameter column and exiting from the top of the 1-inch column. The heating water flow rate was measured for several supply pressures (Table 2).

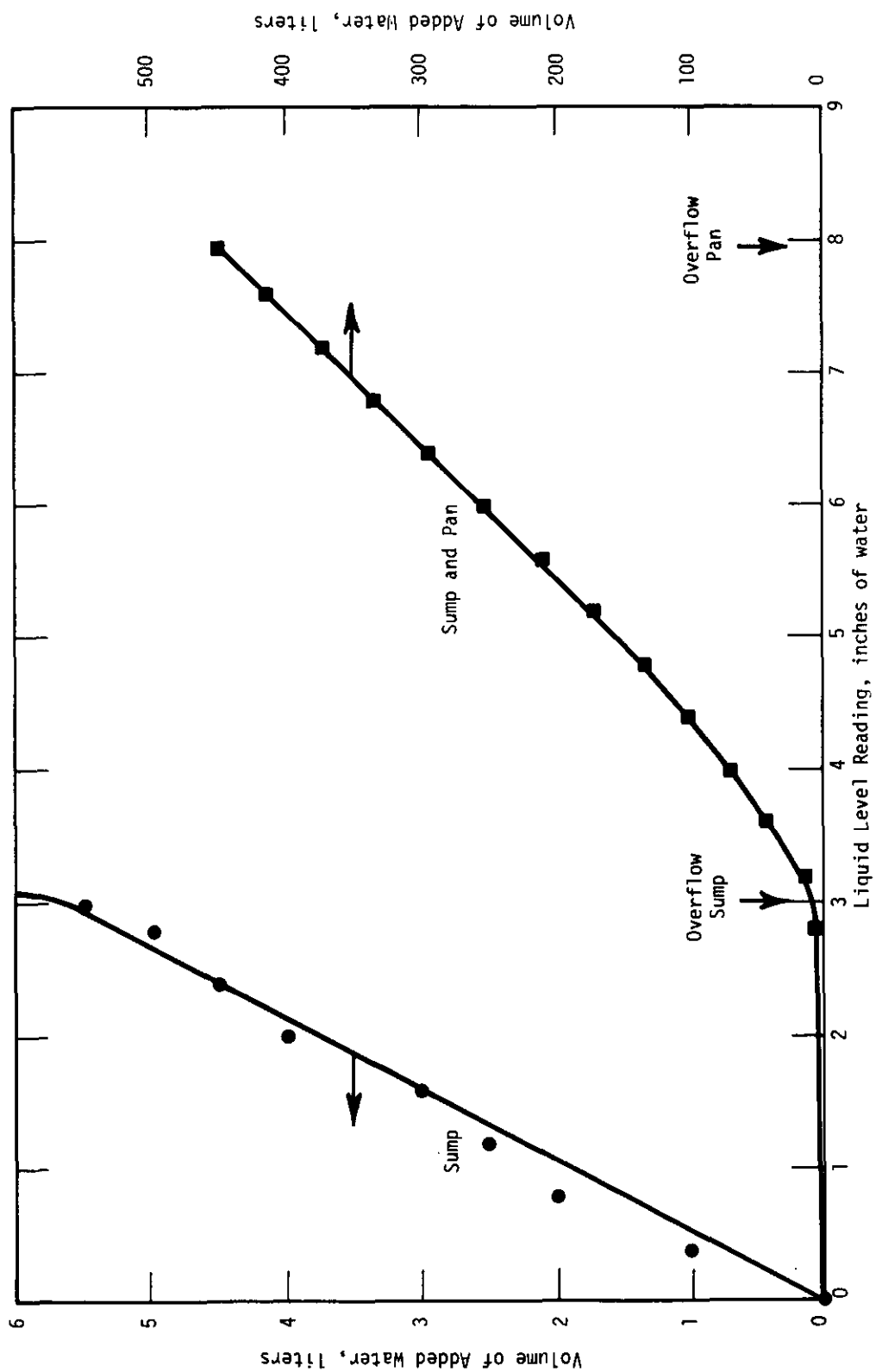


FIGURE 5. Calibration of Sump and Pan

TABLE 1

Cooling Water Flow Rates

Supply Pressure, psig	Supply Temp, °C	Cooling Water Supply Flow Rates, L/min, to				
		RIX-I Evap. 10-1-8 CWR #54	Condenser 10-1-9 CWR #55	RIX-I Feed Tk 10-3-1 CWR #57	RIX-I Waste Tk 10-3-20 CWR #58	Cf RIX-I Run Tk 10-9-1 CWR #64
33.7	35	14.3	—	—	—	—
29.0	36	11.9	11.1	—	—	—
31.3	36	10.0	9.3	8.7	—	—
30.0	36	8.8	8.1	7.8	5.9	—
30.0	39	7.7	7.0	6.4	5.1	7.5
31.5	39	8.7	8.0	7.4	5.7	8.4
30.8	36	13.5	—	—	—	—
31.0	36	—	12.7	—	—	—
31.0	36	—	—	12.0	—	—
32.5	36	—	—	—	9.5	—
30.0	36	—	—	—	—	12.5

TABLE 2

Heating Water Flow Rates

Supply Pressure, psig	Supply Temp, °C	Hot Water Supply Flow to RIX-I Column Jackets HWR #52, L/min
32.5	91.5	10.8
31.1	91.1	10.5
27.2	90.3	10.0
22.3	91.0	9.1
20.1	90.7	8.4

Equipment Tests

Evaporator Liquid-Level Calibration. The accuracy of the lower range of the RIX-I feed adjustment evaporator (EP 10-1-8) volume calibration was verified by addition of small measured amounts of water. The liquid level was read after each addition and the indicated volume was determined from the calibration. The discrepancy between the measured volume and the indicated volume ranged from -2.2% to +8.0% for volumes between 1.25 liters and 15.25 liters (Table 3).

Samplers. The recirculation rate of each sampler was measured by running tubing from the short sample needle into a graduated cylinder containing deionized water (Figure 6) and measuring the time required to remove a given volume of water from the graduate. Actual sampling rates (flow from tank to sampler) were measured by installing a large sample bottle (Figure 7) and measuring the time required to fill the sample bottle with a known volume of deionized water from the tank. Sampler fill and recirculation rates are summarized in Table 4.

The volume of air bleed was so excessive when the bleed line was uncapped that no sample could be obtained. Various size orifices to limit the air bleed rate were tested and the results are shown in Table 5.

The sampler was operable with orifice sizes from 0.0135 to 0.0469 inch; the 0.0312-inch-orifice was selected for all samplers and proved to be satisfactory during all subsequent tests.

Vessel Off-Gas Exhaust System. Figure 8 shows a schematic of the Frame 4 vessel off-gas system. This system is designed to pull enough purge air through the overflow lines of tanks to dilute radiolytically evolved hydrogen to less than 4.0 vol %, the lower explosive limit of hydrogen in air. The system must also have enough capacity to remove internally generated air, such as from spargers and air jet exhausters, in order to prevent expelling air and fumes from any vessel via the overflow line. Off-gas flows were measured at various static pressures for various rates of internal air generation in the condensate tank, RIX-I dump tank, RIX-I waste tank, and Cf RIX-I run tank; results are shown in Appendix C. The design requirements for vessel off-gas exhaust flows (Table 6) were met or exceeded for all tanks at a vacuum of 0.1 inch of water; a vacuum of 0.3 inch of water is available on the off-gas exhaust header.

The RIX-I feed evaporator (EP 10-1-8E) is vented through the condenser (EP 10-1-9) by way of a 2-inch Schedule 40 stainless steel line to the 4-inch Schedule 40 stainless steel off-gas exhaust header. Since the evaporator and condenser are a sealed

TABLE 3

Test of Volume Calibration of Evaporator

Cumulative Volume of Added Water, L	Plant Calibration		% Error
	Liquid Level, inches	Volume, L	
0.25	0.0	0.40	—
1.25	0.95	1.20	-4.0
2.25	1.75	2.43	+8.0
3.25	2.45	3.30	+1.5
4.25	3.15	4.28	+0.7
5.25	3.90	5.35	+1.9
6.25	4.60	6.50	+4.0
7.25	5.30	7.35	+1.4
8.25	6.00	8.25	0.0
9.25	6.70	9.35	+1.1
10.25	7.10	10.25	0.0
11.25	7.40	11.00	-2.2
12.25	7.65	12.25	0.0
13.25	7.80	13.00	-1.9
14.25	8.05	14.13	-0.8
15.25	8.20	15.50	+1.6

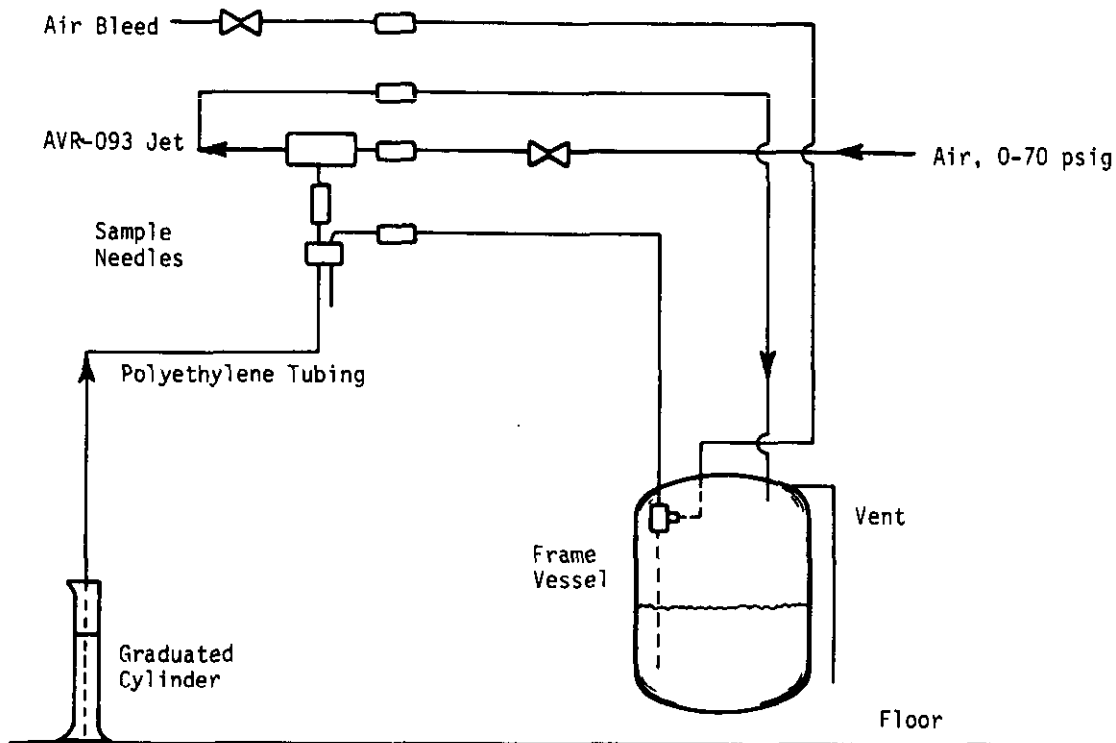


FIGURE 6. Measurement of Sample Recirculation Rate

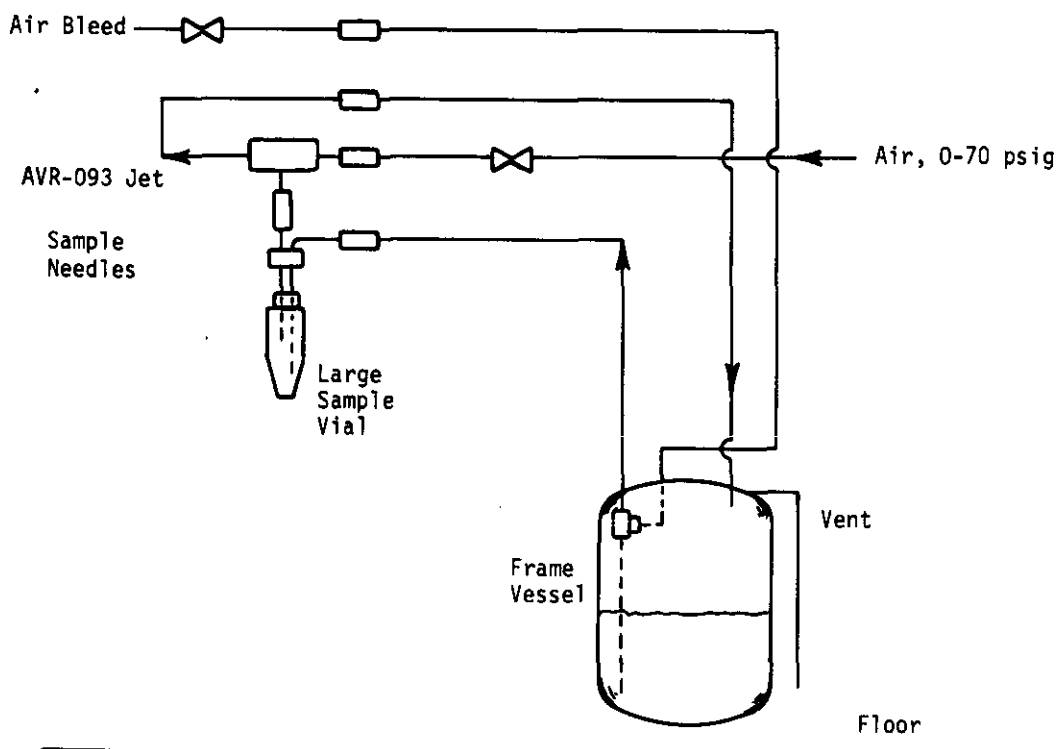


FIGURE 7. Measurement of Sample Fill Rate

TABLE 4

Sampler Fill and Recirculation Rates

<u>Sampler</u>	<u>Fill Rate,^a mL/min</u>	<u>Recirculation Rate, mL/min</u>
10-1-8E	625	250
10-1-10	750	194
10-3-20	1070	250
10-9-1	1500	218

a. With air bleed capped.

TABLE 5

Sampler Air Bleed Orifice Size

<u>Orifice Size, inches</u>	<u>Sample Volume, mL</u>
None	<0.01
0.0938	<0.01
0.0625	<0.01
0.0469	6
0.0312	6
0.0156	8
0.0135	8.5

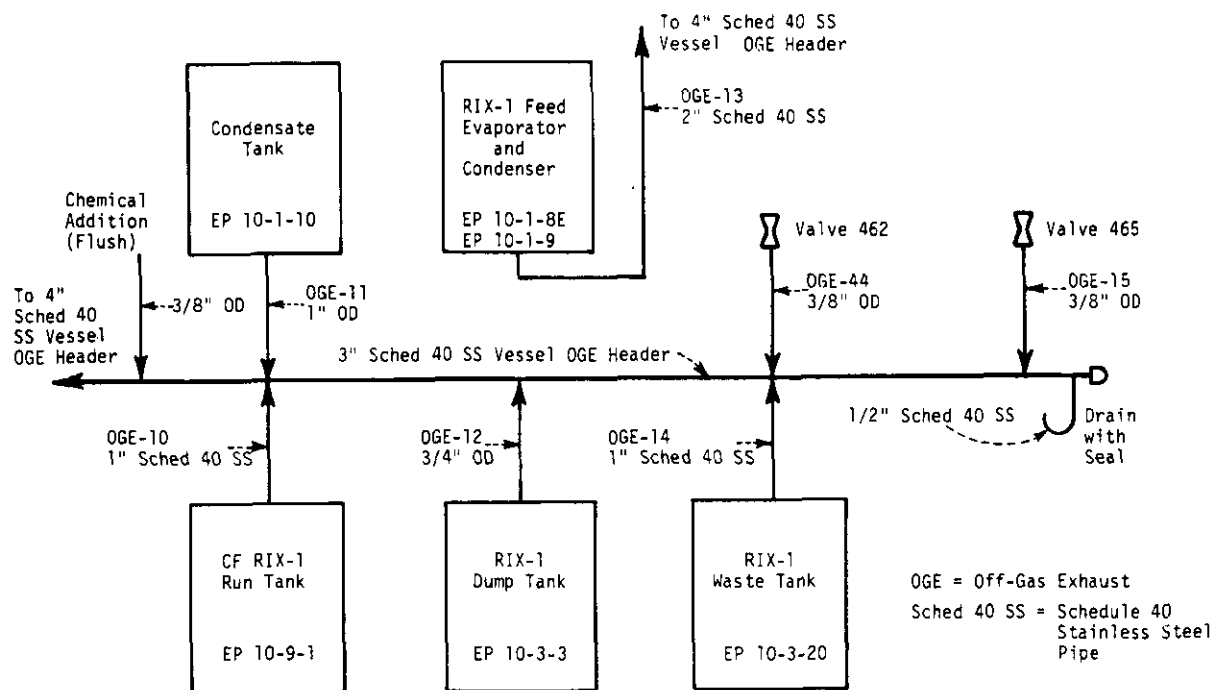


FIGURE 8. Off-Gas Exhaust System for Frame 4 Vessels

TABLE 6

Off-Gas Exhaust Flow Requirements for Frame 4 Vessels

Vessel	EP Number	Internal Air Supplied to Vessel, scfm			Air Purge for Radiolytic Gas Dilution, scfm	Design Basis, scfm
		Transfer Jet	Sample Jet	Sparge		
RIX-I Feed Evap.	10-1-8E	1.67	1.67	1.0	1.0	— ^a
Condensate Tk.	10-1-10	—	1.67	1.0	<0.02	1.67
RIX-I Dump Tk.	10-3-3	—	—	0.75	1.0	1.25
RIX-I Waste Tk.	10-3-20	—	1.67	3.0	0.53	3.75
RIX-I Cf Run Tk.	10-9-1	1.67	1.67	0.75	<0.02	1.67

a. Two-inch Schedule 4 stainless steel exhaust line required to vent off-gas from denitration with formic acid.

system, one scfm of purge air is generated internally and its flow rate is independent of the exhaust system vacuum. Air flows up to 4.9 scfm passed through the system without difficulty.

The 3-inch Schedule 40 stainless steel exhaust header on Frame 4 had a 1/8-inch drain hole at the low point of the header. The 0.3-inch-water static vacuum on the header pulled 0.13 scfm of air in through the hole and prevented complete draining through the hole when flush solution was added to the header. The 1/8-inch drain hole was replaced with a 1/2-inch Schedule 40 stainless steel drain line with a liquid seal to allow complete draining of the header.

Minimum Volume Heater Operation. The minimum liquid level and liquid volume required for operation of the electric heaters in the RIX-I feed adjustment evaporator were determined by tests with water (Table 7). The electric heaters are automatically shut off by a heater high-temperature limit ($>450^{\circ}\text{C}$) if insufficient liquid is present to remove the heat. The minimum liquid level required for operation after the liquid is boiling is less than the minimum liquid level required for startup when the liquid is cold or not boiling. The minimum liquid level for cold startup decreases slightly as the heater voltage is decreased. The minimum liquid level if the liquid is boiling increases slightly as the heater voltage is decreased.

TABLE 7

**Minimum Amount of Liquid^a for Operation
of Heaters in RIX-I Feed Evaporator**

Heater Voltage	Automatic Shutoff During Boildown		Minimum Volume For Startup	
	Level, inches	Volume, L	Level, inches	Volume, L
200	4.3	5.9	6.3	8.8
175	4.3	5.9	6.3	8.8
150	4.4	6.0	6.2	8.5
125	4.7	6.6	6.2	8.5
100	4.9	6.9	6.1	8.4

a. Determined with water.

Heat Transfer to the Evaporator Cooling Coils. The heat transfer rate of the evaporator pot cooling coils was measured by heating 10 liters of water with the electric heaters at 200 volts while cooling water flowed through the coils at 11 liters/minute. At steady state, the inlet cooling water temperature was 38°C, the outlet cooling water temperature was 55.3°C, and the temperature of the water in the evaporator pot was 67°C. From these data, the steady-state heat transfer rate was calculated to be 45,000 Btu/hr and the over-all heat transfer coefficient was calculated to be 310 Btu/(hr)(°F)(ft²).

In a second test, 55 liters of water was heated at 125 volts while cooling with 8.8 liters/minute of cooling water. The 55 liters of evaporator pot liquid was continuously sparged with 15 scfm of air. At steady state, the inlet cooling water temperature was 35°C, the outlet cooling water temperature was 42.4°C, and the temperature of the water in the evaporator pot was 55.2°C. From these data, the steady-state heat transfer rate was calculated to be 15,000 Btu/hr and the over-all heat transfer coefficient was calculated to be 106 Btu/(hr)(°F)(ft²).

Heat Transfer to the Evaporator Condenser. The heat transfer rate of the RIX-I condenser EP 10-1-9 was measured by boiling 10 liters of water in the evaporator with total reflux of the condensate back into the evaporator. Ten liters/minute of cooling water was supplied to the condenser at an inlet temperature of 37°C, with no air sparge or air purge to the evaporator. At steady state, the outlet cooling water temperature was 52.9°C and the vapor temperature of the off-gas at the condenser exit was 40.7°C. From these data, the steady-state heat transfer rate to the condenser cooling water was calculated to be 45,160 Btu/hr; based on a log mean temperature difference of 54.7°C and a heat transfer area of 8.65 ft², the overall heat transfer coefficient was calculated to be 53 Btu/(hr)(°F)(ft²).

In a second test, the boiling water in the evaporator was sparged with 1 scfm of air, which passed through the condenser and out the off-gas exhaust exit. The cooling water inlet temperature and flow rate remained the same as for the previous test. At steady state, the vapor temperature of the off-gas at the condenser exit increased to 42.5°C and the outlet cooling water temperature decreased to 52.4°C. The resultant heat transfer rate to the condenser cooling water decreased slightly, to 43,740 Btu/hr. The over-all heat transfer coefficient decreased slightly, to 51 Btu/(hr)(°F)(ft²). The air leaving the condenser at 42.5°C was saturated with water vapor.

Cooling this saturated air to ambient temperature without dilution with low-humidity air condensed some of the water vapor; about 40 mL/hr of condensate was collected from a low-point drain in the exhaust duct.

Heat Transfer From Feed Tank. The heat transfer rate of the cooling jacket was measured by pumping 2.4 liters/minute of water at 95°C into the RIX-I feed tank. Heat was removed by supplying 10.6 liters/minute of cooling water to the cooling jacket at an inlet temperature of 35°C. At steady state, the outlet temperature of the water from the jacket was 45°C and the outlet temperature of the feed tank water was 57.5°C. From these data, the heat transfer rate to the jacket cooling water was calculated to be 25,000 Btu/hr; based on a log mean temperature difference of 62°F and a cooling jacket heat transfer area of 5.65 ft², the over-all heat transfer coefficient was calculated to be 71 Btu/(hr)(°F)(ft²).

In a second test without any cooling water flowing through the cooling jacket, 2.8 liters/minute of water at 96°C was pumped into the feed tank. The steady-state outlet temperature was 86.5°C. The resultant heat transfer rate from the feed tank to the ambient air was 6,296 Btu/hr; the overall heat transfer coefficient was about 6 Btu/(hr)(°F)(ft²).

Pressure Drops Across Empty Ion-Exchange Columns and Piping. Pressure drops were measured for flow of filtered, deionized water through each empty (no resin bed) RIX-I column and associated piping and valves (Table 8).

The very high pressure drop in both the downflow and upflow directions through the 1-inch column (EP 10-3-13) indicated a severe flow restriction. Subsequent measurements showed that the flow restriction was caused by a defective top frit, as discussed in the next section. The pressure drop for flow through the other columns is somewhat higher than predicted for the empty columns (see below), but is attributed to long lengths of piping and the large number of valves in the process flow path.

Pressure Drops Across Empty Ion-Exchange Columns Only. The columns were removed from Frame 4 and replaced with short pieces of pipe. Each column was placed into the resin loading station for flow testing of the filter frits in the resin retainer plates at the top and bottom of each column.

TABLE 8

Measured Flow Rates and Pressure Drops for
Empty Ion-Exchange Columns and Associated Piping

Ion Exchange Column(s)	Direction of Flow	Route Through Piping and Process Vessels	Water Flow, L/min	Pressure Drop, psig
4"	Downflow	421, 10-3-2, 424, 10-3-1, 426, 442 10-3-10, 401, 10-3-20	2.0	66 to 130
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-3-20	2.0	32 to 73
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 402, 10-9-1	2.0	75 to 76
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 403, 10-21-1 ^a	2.0	58 to 80
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 404, 10-5-1 ^a	2.0	68 to 82
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 406, 10-3-20	0.47	35 to 70
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 410, 10-9-1	0.40	70 to 97
2"	Downflow	429, 10-3-2, 429, 447, 469, 10-3-12, 409, 10-21-1 ^a	0.48	82 to 109
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 406, 10-3-20	0.49	98 to 113
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 408, 10-5-1 ^a	0.47	80 to 150
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 407, 10-19-1 ^a	0.49	130 to 145
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 414, 10-21-1 ^a	0.48	130 to 215
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 411, 10-3-20	0.53	220 to 280
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 415, 10-9-1	0.49	300 to 580
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^a	0.45	550 to 850
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 412, 10-19-1 ^a	0.092	380 to 1010
1"	Upflow	432, 10-3-2, 430, 431, 10-3-13, 470, 407, 10-19-1 ^a	0.52	310 to 1010
2"	Upflow	432, 10-3-2, 448, 471, 10-3-12, 470, 10-3-12, 468, 404, 10-5-1	0.46	7.8
4"	Upflow	432, 10-3-2, 429, 428, 445, 467, 466, 10-3-10, 442, 426, 10-3-1, 424, 441, 10-3-3	0.51	About 2
3"	Upflow	432, 10-3-2, 429, 447, 469, 468, 10-3-11, 466, 401, 10-3-20	0.46	6 to 16
4", 3"	Downflow	432, 10-3-2, 429, 428, 427, 442, 10-3-10, 466, 10-3-11, 405, 10-3-20	1.25	99 to 104

TABLE 8 (Cont'd)

<u>Ion Exchange Column(s)</u>	<u>Direction of Flow</u>	<u>Route Through Piping and Process Vessels</u>	<u>Water Flow, L/min</u>	<u>Pressure Drop, psig</u>
4", 3", 2"	Downflow	432, 10-3-2, 429, 428, 427, 442, 10-3-10, 466, 10-3-11, 468, 10-3-12, 406, 10-3-20	0.33	27 to 44
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.022 to 0.036	20
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^a	0.048 to 0.067	100 to 119
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.068 to 0.075	210 to 216
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.128 to 0.142	365 to 400
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.210 to 0.218	520 to 590
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.298 to 0.300	740 to 795
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 411, 10-3-20 ^b	0.200 to 0.400	660 to 830
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 415, 10-9-1 ^b	0.240 to 0.440	715 to 980
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 414, 10-21-1 ^{a,b}	0.220 to 0.400	780 to 1000
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 413, 10-5-1 ^{a,b}	0.200 to 0.360	910 to 1000
1"	Downflow	432, 10-3-2, 448, 471, 10-3-13, 412, 10-19-1	0.240 to 0.320	1000
1"	Upflow	432, 10-3-2, 430, 431, 10-3-13, 470, 407, 10-19-1 ^{a,b}	0.400 to 0.514	82 to 110
1"	Upflow	433, 10-3-2, 430, 431, 10-3-13, 470, 408, 10-5-1 ^{a,b}	0.400 to 0.480	84 to 130
1"	Upflow	432, 10-3-2, 430, 431, 10-3-13, 470, 409, 10-21-1 ^{a,b}	0.400 to 0.466	114 to 170
1"	Upflow	432, 10-3-2, 430, 431, 10-3-13, 470, 410, 10-9-1 ^b	0.300 to 0.500	180 to 225
1"	Upflow	432, 10-3-2, 430, 431, 10-3-13, 470, 406, 10-3-20 ^b	0.400	235 to 260

TABLE 8 (Cont'd)

Ion Exchange Column(s)	Direction of Flow	Route Through Piping and Process Vessels	Water Flow, L/min	Pressure Drop, psig
4"	Downflow	421, 10-3-2, 425, 442, 10-3-10, 401 ^b	1.7 to 2.2	225
4"	Downflow	421, 10-3-2, 425, 442, 10-3-10, 401 ^b	1.4 to 1.6	210
4"	Downflow	421, 10-3-2, 425, 442, 10-3-10, 401 ^b	1.1 to 1.2	180
4"	Downflow	421, 10-3-2, 425, 442, 10-3-10, 401 ^b	0.6 to 0.88	100 to 128
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-5-1 ^{a,b}	1.94 to 2.1	92 to 94
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-5-1 ^{a,b}	1.34 to 1.96	64 to 72
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-5-1 ^{a,b}	1.2 to 1.4	44 to 50
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-5-1 ^{a,b}	0.58 to 1.08	10 to 29
3"	Downflow	421, 10-3-2, 425, 427, 445, 467, 10-3-11, 405, 10-5-1 ^{a,b}	0.3 to 0.4	9 to 10
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 409, 10-21-1 ^{a,b}	0.46	13 to 19
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 409, 10-21-1 ^{a,b}	0.38	12 to 15
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 409, 10-21-1 ^{a,b}	0.28	10
2"	Downflow	432, 10-3-2, 429, 447, 469, 10-3-12, 409, 10-21-1 ^{a,b}	0.28	6 to 8

a. Simulated by 50-liter bottle since tank is not on this frame.

b. About 90°C hot water supply to jackets.

The maximum pressure drop for water flowing through both frits in series (Table 9) was calculated by Equation 1, which takes into account the porosity, thickness, and area of the frits:

$$\Delta P_{\max} = aF^{1.026} \quad (1)$$

where ΔP_{\max} = maximum pressure drop, psig

F = water flow at ambient temperature, liters/minute

a = a constant for each column

= 11.37 for 4-inch column

= 22.54 for 3-inch column

= 78.05 for 2-inch column

= 300.02 for 1-inch column

TABLE 9

Calculated Maximum Pressure Drops for
Empty Columns with Two Frits in Series

Column Size	Constant "a"	Nominal Water Flow, L/min ^a	Calculated ΔP_{\max} , psig
4"	11.37	1.20	13.7
3"	22.54	0.681	15.2
2"	78.05	0.305	23.1
1"	300.02	0.089	25.1

a. Based on a flow of 16 mL/(min)(cm²).

All RIX-I columns are disposable and must be replaced periodically by new columns. Only those new columns whose measured pressure drops do not exceed the ΔP_{\max} values listed in Table 9 will be accepted.

The maximum pressure drop for water flowing through a single frit is about half that calculated by Equation 1. Measured and calculated pressure drops for empty columns containing only one frit are compared in Table 10 for several flow rates.

The data in Table 10 show that the previously mentioned severe flow restriction of the 1-inch column is in the top frit. The top frit was replaced and the column was retested with down-flow of filtered, deionized water through both frits. The results (Table 11) show that the pressure drop across the 1-inch column was acceptable after the top frit was replaced.

Pressure Drops Through Piping Only. Pressure drops were measured for the flow of filtered, deionized water through the piping and valves without the columns (Table 12). The columns had been removed for off-frame testing of filter frits, as discussed above, and had been replaced by short pieces of pipe.

Pressure Drops Across Resin-Loaded Ion-Exchange Columns. The 4-inch and 3-inch columns were loaded with 100 +20 micron Dowex® 50W-X8 (Dow Chemical Co.) cation-exchange resin; the 2-inch and 1-inch columns were loaded with 35 +15 micron Dowex® 50W-X8 resin. Uniform particle sizes were obtained by hydraulic classification of -200, +400 mesh resin.

Each loaded column was then flow tested in the resin loading station, conditioned with 0.5 M $\text{Zn}(\text{NO}_3)_2$, and flushed with water before installation in Frame 4. Pressure drops were measured during flow testing and conditioning (Table 13).

The resin-loaded columns were then removed from the resin loading station, installed in Frame 4, and flow-tested with filtered, deionized water. Results are shown in Table 14.

The data in Tables 13 and 14 show that the pressure drops are acceptably low. The columns were now ready for the first chemical run following the temperature measurements described below.

Temperatures in Resin-Loaded Ion-Exchange Columns. Solutions entering each of the RIX-I columns must be preheated to about 70°C for efficient operation. The columns are maintained at this temperature by hot water flowing through the column jackets. But the process solution cools while flowing through the long, uninsulated monitor loops between the outlet of one column and the inlet of the

TABLE 10

Measured Pressure Drop Compared to Calculated Maximum
Pressure Drop for Columns with One Frit and No Resin

<u>Column Size</u>	<u>Frit</u>	<u>Direction of Flow</u>	<u>Water Flow, L/min</u>	<u>Measured Pressure Drop, psig</u>	<u>Calculated P_{max}, psig</u>
4"	Top	Downflow	2.0	220	11.6
4"	Top	Downflow	1.4	205	8.0
4"	Top	Downflow	0.9	170	5.1
4"	Top	Downflow	0.5	104	2.8
4"	Top	Upflow	2.8	26.5	16.3
4"	Top	Upflow	2.35	20	13.7
4"	Top	Upflow	1.3	14	7.4
4"	Top	Upflow	1.35	10	7.7
4"	Top	Upflow	0.63	5	3.5
4"	Bottom	Downflow	2.25	27	13.1
4"	Bottom	Downflow	1.45	22	8.3
4"	Bottom	Downflow	1.15	14	6.6
4"	Bottom	Downflow	0.58	7	3.3
3"	Top	Downflow	2.6	80	30
3"	Top	Downflow	2.18	76	25.1
3"	Top	Downflow	1.6	58	18.3
3"	Top	Downflow	1.06	36	12.0
3"	Top	Downflow	0.48	12	5.3
3"	Top	Upflow	2.80	30	32.4
3"	Top	Upflow	2.30	22	26.5
3"	Top	Upflow	1.86	14	21.3
3"	Top	Upflow	1.27	4	14.4
3"	Top	Upflow	0.54	3	6.0
3"	Bottom	Downflow	2.87	23 to 30	33.2
3"	Bottom	Downflow	2.35	20	27.1
3"	Bottom	Downflow	1.80	16	20.6
3"	Bottom	Downflow	1.22	10	13.8
3"	Bottom	Downflow	0.58	5	6.4

TABLE 10 (Cont'd)

<u>Column Size</u>	<u>Frit</u>	<u>Direction of Flow</u>	<u>Water Flow, L/min</u>	<u>Measured Pressure Drop, psig</u>	<u>Calculated ΔP_{max}, psig</u>
2"	Top	Downflow	1.2	45	47.1
2"	Top	Downflow	1.7	64	67.3
2"	Top	Downflow	2.1	82	83.5
2"	Bottom	Downflow	0.6	5	23.1
2"	Bottom	Downflow	0.45	6.5	17.2
2"	Bottom	Downflow	2.3	27	91.7
1"	Top	Downflow	0.22	960	31.7
1"	Top	Downflow	0.30	980	43.6
1"	Top	Downflow	0.25	1000	36.2
1"	Top	Downflow	0.16	960	22.9
1"	Top	Downflow	0.10	450 to 495	14.1
1"	Top	Downflow	0.10	330	14.1
1"	Top	Upflow	0.38	530	55.6
1"	Top	Upflow	0.46	400	67.6
1"	Top	Upflow	0.30	500	43.6
1"	Top	Upflow	0.20	570	28.8
1"	Top	Upflow	0.18	425	25.8
1"	Top	Upflow	0.08	290	11.2
1"	Top	Upflow	0.08	341	11.2
1"	Bottom	Downflow	0.32	185	46.6
1"	Bottom	Downflow	0.18	134	25.8
1"	Bottom	Downflow	0.08	62	11.2
1"	Bottom	Downflow	0.5	141	73.7
1"	Bottom	Downflow	0.4	195	58.6

TABLE 11

Pressure Drop Across Empty One-Inch Column
After Replacement of Top Frit

<u>Water Flow, L/min</u>	<u>Measured Pressure Drop, psig</u>	<u>Calculated ΔP_{max}, psig</u>
0.5	62 to 64	147
0.4	50 to 51	117
0.3	35 to 40	87
0.2	15 to 16	57
0.1	22 to 24	28

TABLE 12

Measured Flow Rates and Pressure Drops for Piping and Valves Only^a

Route Through Piping and Process Vessels	Water Flow, L/min	Measured Pressure Drop, psig
421, 10-3-2, 424, 10-3-1, 426, 442, 401, 10-3-20	2.60	34 to 52
421, 10-3-2, 425, 442, 401, 10-3-20	2.54	56 to 59
421, 10-3-2, 425, 442, 466, 405, 10-3-20	2.33	77 to 79
432, 10-3-2, 429, 428, 427, 442, 466, 405, 10-3-20	2.64	88 to 93
432, 10-3-2, 429, 428, 427, 442, 466, 468, 406, 10-3-20	2.50	110
432, 10-3-2, 429, 428, 427, 442, 466, 468, 470, 411, 10-3-20	2.72	50 to 52
432, 10-3-2, 429, 428, 427, 445, 467, 403, 10-5-1 ^b	2.65	50 to 54
432, 10-3-2, 429, 428, 427, 445, 467, 403, 10-21-1 ^b	2.62	50 to 54
432, 10-3-2, 429, 428, 427, 445, 467, 402, 10-9-1	2.66	52 to 54
432, 10-3-2, 429, 447, 469, 407, 10-19-1 ^b	2.70	48 to 50
432, 10-3-2, 429, 447, 469, 408, 10-5-1 ^b	2.63	50 to 52
432, 10-3-2, 429, 447, 469, 409, 10-21-1 ^b	2.62	48 to 51
432, 10-3-2, 429, 447, 467, 410, 10-9-1	2.62	50 to 52
432, 10-3-2, 448, 471, 411, 10-3-20	2.40	65 to 70
432, 10-3-2, 448, 471, 415, 10-9-1	2.50	49 to 50
432, 10-3-2, 448, 471, 414, 10-21-1 ^b	2.50	48
432, 10-3-2, 448, 471, 413, 10-5-1 ^b	2.50	48 to 50
432, 10-3-2, 448, 471, 412, 10-19-1 ^b	2.5	49 to 50
432, 10-3-2, 430, 431, 470, 407, 10-19-1 ^b	2.5	37 to 38

TABLE 12 (Cont'd)

<u>Route Through Piping and Process Vessels</u>	<u>Water Flow, L/min</u>	<u>Measured Pressure Drop, psig</u>
432, 10-3-2, 430, 431, 470, 408, 10-5-1 ^b	2.5	36 to 38
432, 10-3-2, 430, 431, 470, 409, 10-21-1 ^b	2.73	37 to 48
432, 10-3-2, 430, 431, 470, 410, 10-9-1	2.6	37 to 38
432, 10-3-2, 430, 431, 470, 406, 10-3-20	2.6	36
432, 10-3-2, 429, 428, 427, 442, 466, 468, 470, 412, 10-19-1 ^b	2.53	400

a. Columns removed from Frame 4 and replaced by short pieces of pipe.

b. Simulated by 50-liter bottle since tank is not on Frame 4.

TABLE 13

Pressure Drops Across Resin-Loaded RIX-I Columns in Loading Station

<u>Column</u>	<u>Pressure Drop Measured During</u>	<u>Flow Rate, L/min</u>	<u>Pressure Drop, psig</u>
4"	Water flow through H ⁺ form of resin	1.5	250
3"	Water flow through H ⁺ form of resin	0.9	140
2"	Water flow through H ⁺ form of resin	0.4	550
1"	Water flow through H ⁺ form of resin	0.083	210
4"	Conditioning with 0.5M Zn(NO ₃) ₂	1.5	160
3"	Conditioning with 0.5M Zn(NO ₃) ₂	0.8	140
2"	Conditioning with 0.5M Zn(NO ₃) ₂	0.5	760
1"	Conditioning with 0.5M Zn(NO ₃) ₂	0.08	210
4"	Water flow through Zn ²⁺ form of resin	1.5	160
3"	Water flow through Zn ²⁺ form of resin	0.85	140
2"	Water flow through Zn ²⁺ form of resin	0.51	810
1"	Water flow through Zn ²⁺ form of resin	0.09	210

TABLE 14

Pressure Drops Across Resin-Loaded RIX-I Columns in Frame 4

<u>Flow Path Through Columns in Series</u>	<u>Water Flow, L/min</u>	<u>Water Temperature at Outlet from Last Column, °C</u>	<u>Maximum Pressure Drop, psig</u>
4"	1.2	76	135
4" and 3"	0.7	77	175
4", 3", and 2"	0.4	78	580
4", 3", 2", and 1"	0.09	80	429

next during series operation of the columns. This heat loss is most severe at the lower flow rates to the smaller columns. The solution is therefore preheated before entering the two smaller columns by flowing through coils wrapped around the outside of the hot water jacket of the 1-inch and 2-inch columns.

Temperatures of the "feed solution" (deionized, filtered water) were measured at the inlet to the 4-inch and 3-inch columns, at the inlet to the preheater coils of the 2-inch and 1-inch columns, and at the outlets from all four columns, while the feed water passed through various combinations of columns in series at the nominal flow rates. During these tests, 2.5 gal/min of hot water was supplied to the column jackets in series, at a pressure of 31.5 psig. The temperature of the hot water ranged from 90 to 93°C at the inlet to the 4-inch-column jacket, and from 86 to 90°C at the outlet from the 1-inch-column jacket. Measured temperatures and flow rates are listed in Table 15.

The "feed solution" was inadequately preheated by the coils around the column jackets because of:

- Lack of adherence of the solid heat-transfer medium between the jacket and the coils;
- Excessive heat losses from the outlet line of the coils.
- Insufficient heat-transfer area.

Examination of the heat-transfer medium revealed that regular or water-soluble (Type S) material (Zeston® Grade Z-10, Zeston Company) had been used instead of the specified high-strength waterproof (Type IS) material. The Type S material had cracked and crumbled severely, allowing loss of contact with the jacket wall in many places, thus reducing the effective heat-transfer

TABLE 15

Temperature of Feed Water Entering and Leaving
Resin-Loaded Columns During Heating Tests

Flow Path Through Columns in Series	Water Flow, L/min	Water Temperature, °C							
		4" Column		3" Column		2" Column		1" Column	
		In	Out	In	Out	In	Out	In	Out
4"	1.2	55 ^a	71	-	-	-	-	-	-
4" and 3"	0.7	53 ^a	75	62	76	-	-	-	-
4", 3", and 2"	0.4	34 ^a	77	58	79	59	78	-	-
4", 3", 2", and 1"	0.07	34 ^a	77	38	81	38	76	36	72

a. The temperature of the "feed solution" (water) ranged from 81 to 84°C as it left the heated feed tank and entered the monitor loop; by the time the feed water reached the 4-inch column, it had cooled to from 55°C (at the highest flow rate) to 34°C (at the lowest flow rate).

area. The coils were not secured well enough to the jacket wall by the stainless steel bands to prevent movement. Type S material is also unsatisfactory because it is water-soluble and thus easily washed from the column by any process or water leaks.

The coils were also wrapped adjacent to each other, leaving no space for the proper application of the heat-transfer material. Furthermore, the coils were wrapped over only about half of the available jacket area.

The cold inlet line to the coil was in contact with the hot outlet line from the coil, so that the hot "feed solution" was cooled unnecessarily before entering the column. Furthermore, the hot outlet line from the coil at the bottom of the column was allowed to air-cool enroute to the top of the column.

The original preheater coils were later removed and replaced with new coils over the available jacket area. The coils were rearranged so that the "feed solution" flowed from the bottom of the column jacket to the top, thus minimizing the heat losses between the outlet of the preheater coils and the inlet to the column. The coils and hot outlet lines were cemented to the jacket with high-strength, water-proof Type IS heat-transfer material. The "feed solution" was then adequately preheated by the jacket preheater coils. The technique for proper installation of the jacket preheater coils and subsequent heat-transfer tests have been described previously.¹

1. W. J. Jenkins. Project S-1085, 221-F-Multipurpose Processing Facility, Technique for Proper Installation of Jacket Preheater Coils on RIX Columns. DPST-72-336, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, SC (May 3, 1972).

Removal of Resin from Dump Tank. Two tests were made to measure the efficiency of resin removal from the resin dump tank 10-3-3. In both tests, a measured volume of ~200, +400 mesh Dowex® 50W-X8 resin was added to the dump tank and then removed by flushing with 3-liter volumes of deionized water. During the second test, each 3-liter volume was sparged with 1 scfm of air for five minutes through spare dip-leg # sp 252. Results (Table 16) show that although resin removal was more efficient when each flush was air sparged, essentially all of the resin can be removed without air sparging by using more flushes.

TABLE 16

Removal of Resin from Dump Tank

Water Flush (3 liters each)	Amount of Resin Removed by Flushing			
	Without Sparge		With Air Sparge	
	Volume, mL ^a	%	Volume, mL ^b	%
1	500	49.02	750	75.38
2	304	29.80	180	18.09
3	88	8.63	34	3.42
4	43	4.22	15	1.51
5	21	2.06	6.5	0.65
6	14	1.37	3.6	0.36
7	7.0	0.69	1.85	0.19
8	5.5	0.54	<0.1	<0.01
9	1.0	0.10	-	-
10	0.7	0.07	-	-
11	<0.1	<0.01	-	-
Total	984.3	96.50	991.05	99.60

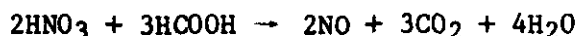
a. Initial amount of resin added = 1020 mL.

b. Initial amount of resin added = 995 mL.

COLD CHEMICAL RUNS

Evaporation, Steam Stripping, and Denitration. The objective of evaporation, steam stripping, and denitration is to reduce the volume of a batch of RIX-I feed to less than 20 liters and the concentration of nitric acid to 1M or less. The usual procedure is to evaporate the batch to about 10 liters, then steam-strip by adding water and boiling until the nitric acid concentration is 4M to 5M, and finally denitrate by adding a measured volume of formic acid at a controlled rate of not more than 25 mL/min. The water used for steam stripping is added below the surface of the feed solution through a dip-tube. Water is added to balance evaporation, so that the volume of feed solution is held constant at about 10 liters. Removal of HNO_3 by stripping is effective at higher acidities. But steam stripping becomes relatively inefficient at concentrations less than about 4M HNO_3 ; more than 4.5 volumes of water are required per volume of feed solution.

Denitration of solutions 1M to about 4M HNO_3 by formic acid proceeds by the following reaction:



When the air purge is about 0.05 scfm or less, 1.5 moles of formic acid are required per mole of nitric acid, according to the above equation. But if the air purge is increased to 1.0 scfm, the required amount of formic acid is increased to between 2.0 and 2.5 moles per mole of nitric acid.

At nitric acid concentrations above 2M, the reaction is rapid; denitration proceeds as fast as the formic acid is added if the addition rate does not exceed 25 mL/min. However, as the nitric concentration decreases to about 2M, the total acidity does not decrease as rapidly as does the nitric acid concentration, which indicates a buildup of formic acid as denitration slows down. Refluxing for about 2 hours after the last addition of formic acid allows denitration to continue; the final concentration of excess formic acid is usually less than about 0.5 to 1.0M. Denitration of nitric acid by formic acid has been described in detail.²

2. R. F. Bradley and C. B. Goodlett. Denitration of Nitric Acid Solutions by Formic Acid. USAEC Report DP-1299, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, SC (1972).

Six cold chemical runs were made in the feed adjustment evaporator. The first five runs consisted of evaporation, steam-stripping, and denitration; the fourth run included a second denitration step, and the sixth run was for denitration only. Runs 4, 5, and 6 used two moles of neodymium nitrate to simulate the actinides and lanthanides in radioactive feed. The total volume of water used for steam-stripping varied from 66 to 139 liters. The total nitric acid remaining in the feed solution after steam stripping was 47.2 to 48.8 moles for Runs 1, 2, and 3 without neodymium nitrate, and 26.7 and 25.7 moles for Runs 4 and 5 with neodymium nitrate. During these five runs, steam-stripping removed enough nitric acid to permit further denitration with formic acid. Table 17 summarizes the analyses of duplicate samples of evaporator bottoms and condensate for these five runs. Appendix D shows individual sample analyses and material balances.

Analyses of neodymium in the evaporator bottoms and condensate (Table 17) were used to calculate entrainment of bottoms solution in the overheads (condensate) during Runs 4 and 5 (Table 18).

The evaporator was purged with 1 scfm air during all six runs. The temperature of the evaporator solution was controlled at $95 \pm 3^\circ\text{C}$ during addition of formic acid at 20 ± 2 mL/min. For Runs 1 and 2, the solution was cooled immediately after completion of the formic acid addition; for all other runs, the solution was refluxed for 2 hours after completion of the formic acid addition before cooling. Total H^+ was determined for all runs; for the last four runs, total NO_3^- was also determined. The total H^+ analyses are summarized in Table 19 in order of decreasing total H^+ concentration before addition of formic acid.

The total NO_3^- analysis are summarized in Table 20 for the last four runs in order of decreasing initial total NO_3^- concentration.

Accuracy of the Evaporator Specific-Gravity Bubbler. Accuracy of the specific-gravity bubbler in the feed adjustment evaporator was verified by taking replicate samples during various chemical runs for laboratory determination of specific gravity. Agreement between laboratory measurements and bubbler measurements (Table 21) indicated satisfactory accuracy.

TABLE 17

Composition of Evaporator Bottoms and Overheads During Evaporation and Steam Stripping

Run	Evaporator Bottoms					Overheads (Condensate)						% of H ⁺ Originally in Evaporator Feed
	Total H ⁺		Total NO ₃ ⁻		Nd(NO ₃) ₃ ^c	Total H ⁺		Total NO ₃ ⁻		Nd(NO ₃) ₃ ^c		
	molarity	moles	molarity	moles		molarity	moles	molarity	moles			
1	11.5	106.4	NA ^a	NA ^a	0	0.39	22.9	NA ^a	NA ^a	0	18	
	5.8	81.2	NA	NA	0	1.7	28.6	NA	NA	0	26	
	7.9	73.1	NA	NA	0	0.57	8.3	NA	NA	0	10	
	5.9	62.0	NA	NA	0	0.45	12.5	NA	NA	0	17	
	4.7	52.2	NA	NA	0	0.26	7.2	NA	NA	0	12	
	4.3	49.5	NA	NA	0	0.11	3.0	NA	NA	0	6	
	4.7	48.8	NA	NA	0	0.21	5.5	NA	NA	0	10	
2	13.1	88.1	NA	NA	0	0.76	46.8	NA	NA	0	35	
	6.1	63.6	NA	NA	0	0.90	28.8	NA	NA	0	31	
	4.6	47.9	NA	NA	0	0.21	7.1	NA	NA	0	13	
3	12.5	93.6	NA	NA	0	0.62	61.6			0	40	
	5.3	54.1	4.8	48.9 ^b	0	1.25	39.5	0.95	30.0 ^b	0	42	
	4.9	47.3	4.3	41.7 ^b	0	0.20	7.0	0.05	1.8 ^b	0	13	
4	12.3	86.4	11.8	83.2	NA ^a	0.74	44.2	NA ^a	NA ^a	<0.00042	34	
	5.2	40.6	6.1	48.0	1.77	0.87	51.5	0.95	56.1	NDC ^c	56	
	4.4	34.8	5.3	42.5	1.92	0.20	7.0	0.22	7.6	<0.00024	17	
	3.8	29.7	3.4	26.4	1.82	0.13	4.5	0.13	4.5	<0.00024	13	
	3.5	26.7	4.4	34.3	1.77	0.10	3.4	0.24	8.0	0.00094	11	
5	11.4	107.4	11.1	104.6	2.82	0.85	44.2	0.87	45.0	NDC ^c	29	
	5.3	41.8	6.2	48.4	1.84	1.33	43.9	1.57	51.7	<0.00023	51	
	4.1	34.3	5.0	41.8	1.88	0.27	9.0	0.36	12.2	0.0014	21	
	3.5	28.9	4.5	36.6	1.87	0.11	4.3	0.18	6.7	<0.00026	13	
	3.1	25.7	4.2	35.1	1.90	0.08	2.6	NA ^a	NA ^a	0.0021	9	

a. Not analyzed.

b. Only HNO₃ present for Run 3, total H⁺ should equal total NO₃⁻.

c. Not detected.

TABLE 18

Entrainment of Evaporator Bottoms in Condensate

$\left(\frac{\text{Total Moles of Nd}^{3+} \text{ in Bottoms}}{\text{Total Moles of Nd}^{3+} \text{ in Condensate}} \right)^a$	% Entrainment
>4800	<0.02
None detected in condensate	—
>8000	<0.013
>8000	<0.013
1900	0.05
>8000	<0.013
1300	0.08
7200	0.014
900	0.11

a. Combined data for Runs 4 and 5.

TABLE 19

Total H⁺ Concentration During Denitration With Formic Acid

Run	Total H ⁺ Concentration in Evaporator Solution ^a				Amount of HCOOH Added	
	Before Addition of HCOOH		After Addition of HCOOH		HCOOH/Initial	Total H ⁺ , mole ratio
	molarity	moles	molarity	moles	moles	
3	4.85	47.3	2.15	21.0	59.6	2.26
1	4.69	48.8	2.32	21.1	61.4	2.22
2	4.61	47.9	1.83	18.4	59.7	2.02
6	3.90	38.5	1.88	19.3	45.0	2.34
4A	3.45	26.7	2.19	16.1	25.0	2.36
5	3.12	25.7	1.10	10.5	41.0	2.68
4B	2.24	15.8	1.36	12.3	11.1	3.16

a. Average of duplicate analyses.

TABLE 20

Total NO₃⁻ Concentration During Denitration With Formic Acid

Run	Total NO ₃ ⁻ Concentration in Evaporator Solution ^a				Amount of HCOOH Added	
	Before Addition of HCOOH		After Addition of HCOOH		HCOOH/Initial	Total NO ₃ ⁻ , mole ratio
	molarity	moles	molarity	moles	moles	
3	4.28	41.7	0.67	6.5	59.6	1.69
6	4.63	45.7	2.40	24.6	45.0	2.13
4A	4.42	34.3	2.59	19.0	25.0	1.64
5	4.25	35.1	1.65	15.6	41.0	2.11
4B	3.05	21.6	1.98	18.0	11.1	3.08

a. Average of duplicate analyses.

TABLE 21

Verification of Accuracy of Evaporator Specific-Gravity Bubbler

Sample Number	Specific Gravity of Various Solutions in Evaporator During Chemical Runs		
	<u>Laboratory Determination</u>		<u>Measured by Evaporator Specific-Gravity Bubbler</u>
	<u>Specific Gravity</u>	<u>Average</u>	
422	1.3999		
423	1.3983	1.399	1.40
447	1.1861		
448	1.2108	1.198	1.20
709	1.1545		
710	1.1703	1.162	1.18
715	1.1414		
716	1.1436	1.143	1.14
719	1.149		
720	1.145	1.147	1.15
730	1.0933		
731	1.0561	1.075	1.08
741	1.2343		
742	1.2358	1.235	1.24
747	1.1953		
748	1.1935	1.194	1.18
751	1.1779		
752	1.1812	1.180	1.18
766	1.0912		
767	1.0946		
777	1.0908		
778	1.0879	1.091	1.08
770	1.1840		
771	1.1782	1.181	1.18
772	1.1116		
773	1.1147		
774	1.1129		
775	1.1094	1.112	1.10

Product Holdup in the Feed Evaporator and Filter. The product leaving the feed adjustment evaporator passes through a porous stainless-steel filter during the normal transfer into the RIX I feed run tank (EP-10-3-1). The contents of the feed run tank can be transferred back into the evaporator via the evaporator transfer tank (EP 10-1-5).

Tests were made using the HNO_3 solutions from the evaporator chemical runs to determine the percentage of the batch received after transfer, and the additional percentage received after each of two water flushes following the transfer. Percentage of each batch received was determined by H^+ analysis and, in some cases, by NO_3^- analysis too. Test results showed that all of the batch was received when followed with two water flushes of 1 to 2 liters. The first water flush yielded an additional 5.2% or more, and the second water flush yielded an additional 1.6 to 3.3%. Only 87.9 to 94.0% of the batch was received when water flushes were omitted. Results are shown in Tables 22 (Run 1), 23 (Run 2), and 24 (Run 3).

The stainless-steel filter in the transfer line between the evaporator and the feed run tank was removed after transfer of Run 5 solution followed by two 3-liter water flushes. Draining the filter yielded 18.5 mL of solution (Sample 779) which contained 0.04M H^+ (about 0.007% of the total H^+ present in Run 5) and 583 $\mu\text{g/mL}$ Nd^{3+} (about 0.004% of the total neodymium).

RIX-I Displacement Chromatography. Californium, curium, and americium are separated from each other and from fission-product lanthanides on the rapid ion-exchange RIX-I columns by displacement chromatography. The RIX-I columns are four 4-foot-long columns with inside diameters of 4, 3, 2, and 1 inch, respectively. All columns have hot-water jackets to control the temperature inside the columns at 70°C or above. The 2-inch and 1-inch columns also have preheater coils to raise the temperature of the entering solutions to 70°C or above. The 4-inch and 3-inch columns were loaded with 100 +20-micron resin which had been hydraulically classified from -200, + 400 mesh Dowex® 50W-X8 cation-exchange resin. The 2-inch and 1-inch columns were loaded with 35 +15-micron resin which had been hydraulically classified from -200, + 400-mesh Dowex® 50W-X8 resin. Resins were saturated with zinc before each chemical run.

Nuclides are separated by displacement chromatography with 0.05M DTPA (diethylenetriaminepentaacetic acid) complexing anion as a developing agent. The DTPA is adjusted to pH 6 with ammonium hydroxide. The actinides and lanthanides have a higher affinity for the resin than the zinc, so they displace the zinc as they are loaded onto the top half of the resin of the 4-inch column. The DTPA developing agent is fed downflow through the column and displaces the actinides and lanthanides which, in turn, displace zinc

TABLE 22

Feed Evaporator and Filter Transfer Heel for Run 1

Solution Analyzed	Solution Volume, L	Sample Number	Total H ⁺			Total NO ₃ ⁻			Material Balance, %	
			molarity	moles	% ^a	molarity	moles	% ^a	H ⁺	NO ₃ ⁻
Solution initially in feed adjustment evaporator (EP 10-1-8E)	9.1	34 35	2.32 2.32	21.11	100	NA ^b	NA ^b	-	-	-
Solution received in evaporator transfer tank (EP 10-1-5) after transfer from evaporator through filter via feed run tank (EP 10-3-1)	8.0	36 37	2.32 2.32	18.56	87.9	NA	NA	-	87.9	-
First one-liter water flush of evaporator and filter received in evaporator transfer tank	1.2	38 39	2.32 2.32	5.38	25.5	NA	NA	-	113	-
Second one-liter water flush of evaporator and filter received in evaporator transfer tank	1.0	40 41	0.69 0.69	0.69	3.3	NA	NA	-	117	-
Total received in evaporator transfer tank				24.63	117					

a. Percent of initial total in evaporator solution.

b. Not analyzed.

TABLE 23

Feed Evaporator and Filter Transfer Heel for Run 2

Solution Analyzed	Solution Volume, L	Sample Number	Total H^+			Total NO_3^-			Material Balance, %	
			molarity	moles	% ^a	molarity	moles	% ^a	H^+	NO_3^-
Solution initially in feed adjustment evaporator (EP 10-1-8E)	10.1	57 58	1.85 1.80	18.43	100	NA ^b	NA ^b	-	-	-
Solution received in evaporator transfer tank (EP 10-1-5) after transfer from evaporator through filter via feed run tank (EP 10-3-1)	9.63	59 60	1.86 1.74	17.33	94.0	1.16 1.20	11.36	-	94.0	-
First one-liter water flush of evaporator and filter received in evaporator transfer tank	2.0	61 62	0.42 0.53	0.95	5.2	0.18 0.17	0.35	-	99.2	-
Second one-liter water flush of evaporator and filter received in evaporator transfer tank	2.0	63 64	0.11 0.19	0.30	1.6	0.02 0.02	0.04	-	101	-
Total received in evaporator transfer tank				18.58	101		11.75			

a. Percent of initial total in evaporator solution.

b. Not analyzed.

TABLE 24

Feed Evaporator and Filter Transfer Heel for Run 3

Solution Analyzed	Solution Volume, L	Sample Number	Total H ⁺			Total NO ₃ ⁻			Material Balance, %	
			molarity	moles	% ^a	molarity	moles	% ^a	H ⁺	NO ₃ ⁻
Solution initially in feed adjustment evaporator (EP 10-1-8E)	9.75	85	2.15	20.96	100	0.50	6.48	100	-	-
		86	2.15			0.83				
Solution received in evaporator transfer tank (EP 10-1-5) after transfer from evaporator through filter via feed run tank (EP 10-3-1)	9.0	87	2.15	19.58	93.4	0.91	8.01	124	93.4	124
		88	2.20			0.87				
First one-liter water flush of evaporator and filter received in evaporator transfer tank	1.80	89	0.70	1.23	5.9	0.034	0.06	0.9	99.3	125
		90	0.67			0.033				
Second one-liter water flush of evaporator and filter received in evaporator transfer tank	1.88	91	0.185	0.38	1.8	ND ^b	-	-	101	125
		92	0.148			ND				
Total received in evaporator transfer tank				21.19	101		8.07	125		

a. Percent of initial total in evaporator solution.

b. Not detected.

as each moves down the column in a zone. The DTPA complexes of these elements differ significantly in stability; consequently, the elements separate into individual bands within the zone, with californium in front, followed by curium and americium and finally by the lanthanide fission products. NH_4^+ is retained by the resin at the rear boundary of the zone and the zone itself is composed essentially of actinides and lanthanides complexed by the DTPA. After a certain column length, the bands attain their steady-state "shape". The bands follow one another without intervals and move at equal rates. This displacement type of development, therefore, results in finite overlap regions of fixed length between neighboring bands which cannot be avoided nor reduced in length.

Further increase in column length (at constant column diameter) does not improve separation. To further improve separation, the bands are passed through successive columns with smaller diameters; the overlap regions have the same length as in large column, but contain less actinides in the overlap regions.

Two chemical runs were made in the RIX-I columns using a mixture of nonradioactive lanthanides (dysprosium, terbium, gadolinium, europium, samarium, and neodymium) to simulate the separation of actinides and fission-product lanthanides. The same resins were used for both runs by flushing between runs with excess DTPA to remove all traces of lanthanides and then reconditioning with 0.5M $\text{Zn}(\text{NO}_3)_2$ to saturate the resin with zinc. A total of 4.0 moles of lanthanides was loaded and eluted from the columns in the first run and a total of 2.5 moles of lanthanides was loaded and eluted from the columns in the second run. The entire effluent from the 1-inch column was collected in 1-liter composite samples for each run. The make-up compositions of the feed solutions are given in Appendix E. Composite sample analyses are shown in Appendix F and the elution profiles are shown in Figures 9 and 10 for Runs 1 and 2. These profiles demonstrate satisfactory separation of the lanthanides by the RIX-I columns.

Performance of the RIX-I columns and associated equipment in Frame 4 was satisfactory. The minimum, average, and maximum column operating pressures for each 190 minutes of elution during Runs 1 and 2 are summarized in Table 25. The column temperatures during Runs 1 and 2 are summarized in Table 26.

The temperature of the hot water supply to the column water jackets ranged from 85 to 95°C during Run 1 and from 85 to 94°C during Run 2. The temperature of the hot water return from the column water jackets ranged from 83 to 94°C during Run 1 and from 85 to 92°C during Run 2. The temperature of the DTPA elutriant, which was preheated before pumping to Frame 4, ranged from 81 to 99°C during Run 1 and from 76 to 85°C during Run 2.

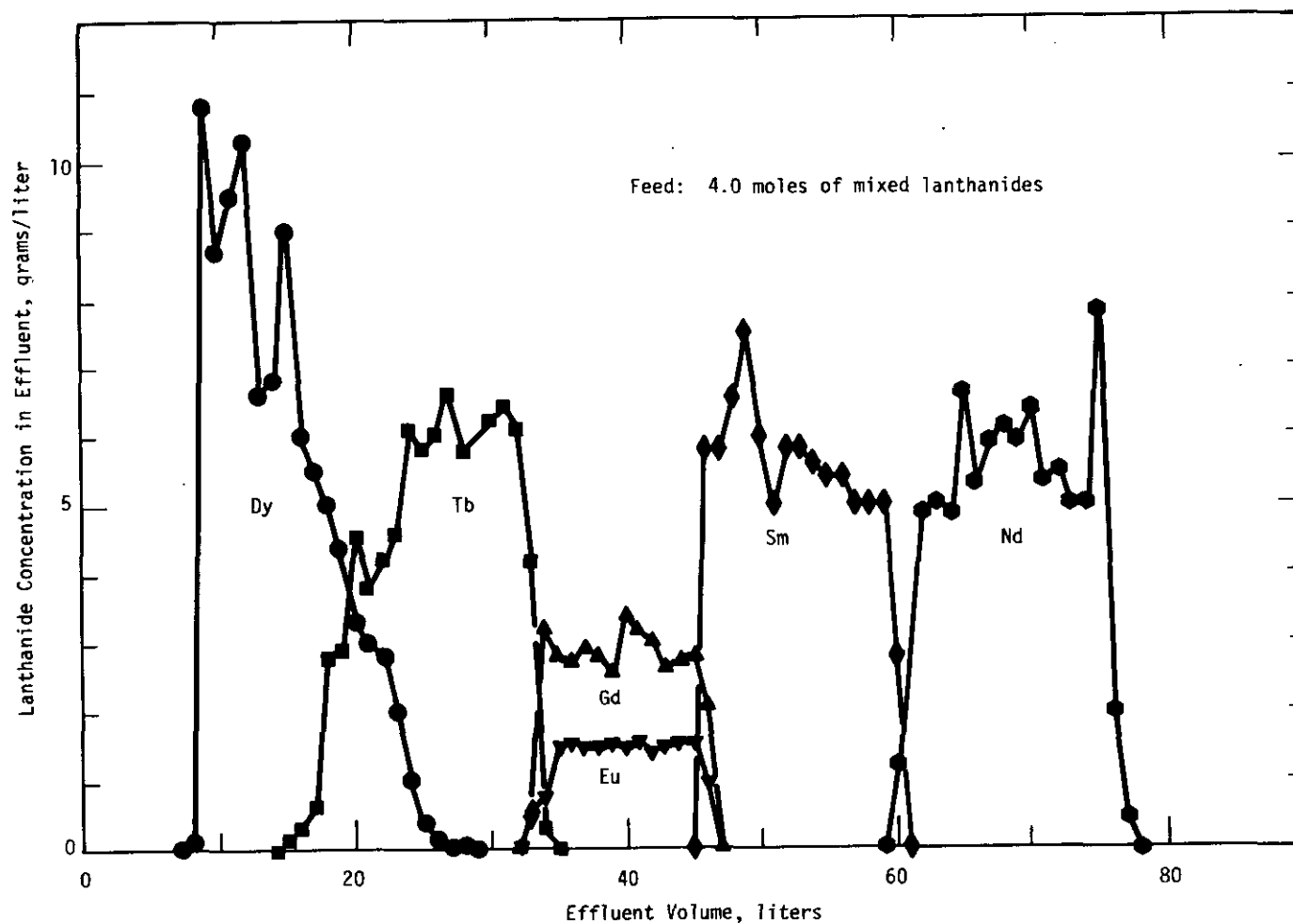


FIGURE 9. Elution Profiles for Run 1 with Nonradioactive Lanthanides in RIX-I Columns

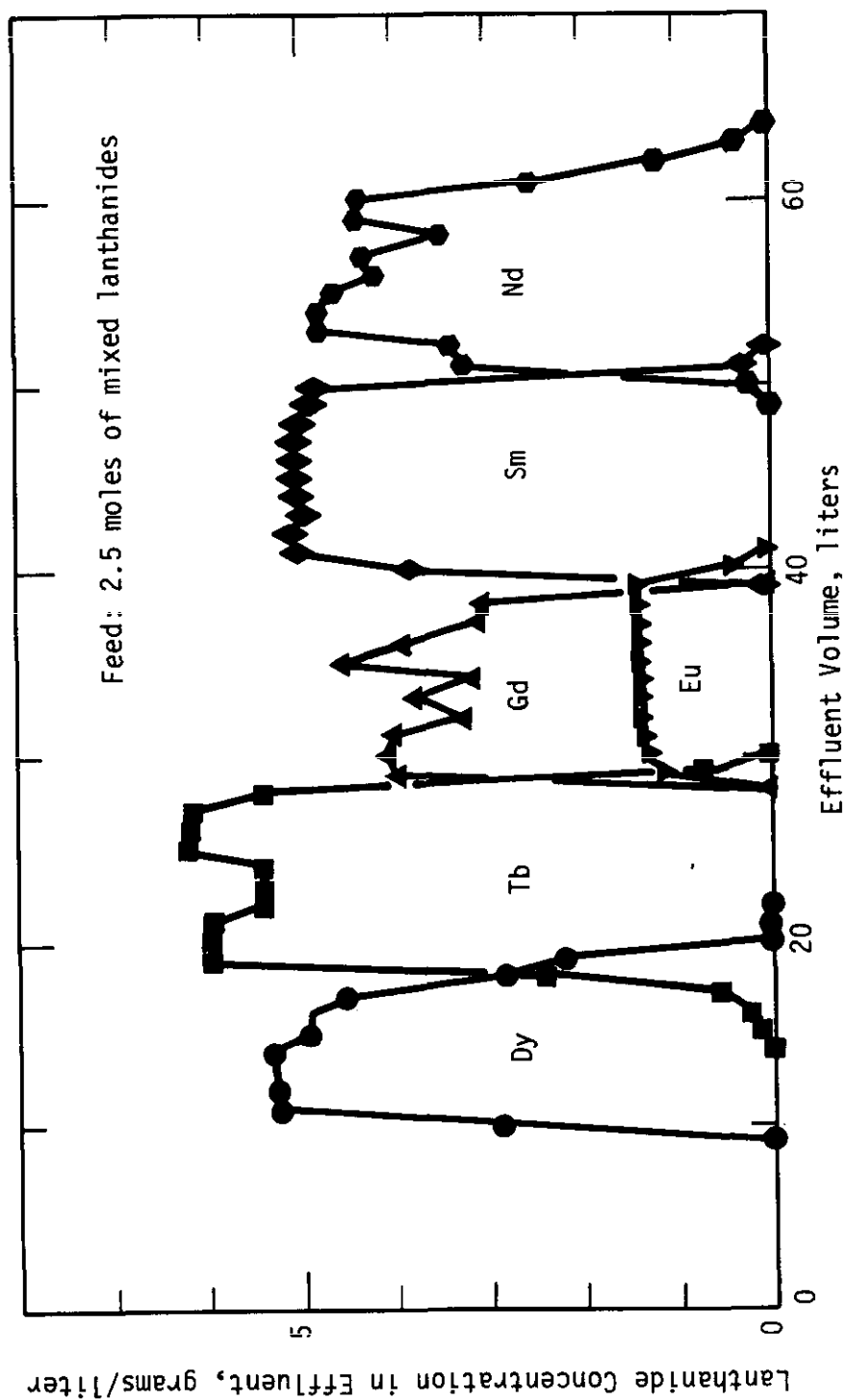


FIGURE 10. Elution Profiles for Run 2 with Nonradioactive Lanthanides in RIX-I Columns

TABLE 25

RIX-I Column Operating Pressures During Nonradioactive Chemical Runs

Run Number	Flow Path Through Columns	Column Operating Pressures			Average Flowrate, L/min
		Minimum, psig	Average, psig	Maximum, psig	
1	4" Feed Displacement	260	866	1000	0.75
	4" Elution	110	138	190	1.12
	4" and 3"	135	163	185	0.92
	4", 3", and 2"	450	467	475	0.44
	4", 3", 2", and 1"	100	281	390	0.145
	4", 3", 2", and 1"	390	393	400	0.154
	4", 3", 2", and 1"	380	433	450	0.142
	4", 3", 2", and 1"	450	513	630	0.138
2	4" Feed Displacement	50	106	118	1.15
	4" Elution	90	90	90	1.25
	4" and 3"	120	124	135	0.80
	4", 3", and 2"	650	660	670	0.45
	4", 3", 2", and 1"	470	511	540	0.07
	4", 3", 2", and 1"	320	391	500	~0.1
	4", 3", 2", and 1"	360	379	410	~0.1
	4", 3", 2", and 1"	300	436	600	~0.1
	4", 3", 2", and 1"	220	718	970	~0.1

TABLE 26

RIX-I Column Temperatures During Nonradioactive Chemical Runs

Run Number	Flow Path Through Columns	Column Temperatures, °C								Average Flowrate, L/min
		4" Column		3" Column		2" Column		1" Column		
		In	Out	In	Out	In	Out	In	Out	
1	4" Feed Displacement	28	70							0.75
	4" Elution	46	71							1.12
	4" and 3"	43	73	63	76					0.92
	4", 3", and 2"	38	73	56	78	55	78			0.44
	4", 3", 2", and 1"	29	79	37	80	36	78	35	79	0.145
	4", 3", 2", and 1"	30	79	38	80	36	80	35	81	0.154
	4", 3", 2", and 1"	27	72	35	75	34	75	32	75	0.142
	4", 3", 2", and 1"	28	75	35	77	32	77	30	79	0.138
2	4" Feed Displacement	28	70							1.15
	4" Elution	55	74							1.25
	4" and 3"	52	75	62	77					0.80
	4", 3", and 2"	47	76	58	79	60	78			0.45
	4", 3", 2", and 1"	33	77	38	79	38	78	37	79	0.07
	4", 3", 2", and 1"	35	78	38	80	38	78	37	79	0.1
	4", 3", 2", and 1"	35	78	38	80	39	80	38	80	0.1
	4", 3", 2", and 1"	28	75	32	79	32	77	31	77	0.1
	4", 3", 2", and 1"	28	70	28	74	28	74	28	73	0.1

The actinide-lanthanide feed solution is displaced from the RIX-I column feed tank into the columns by pumping water into the feed tank. The feed tank is baffled to induce plug flow for efficient removal of the feed solution with a minimum displacement volume of water. For both Runs 1 and 2, the feed solution was displaced by 60 liters of water. The residual solution in the column feed tank after each run was mixed and analyzed to determine the percent of feed which remained in the feed tank after displacement by water. The percent feed remaining in the tank residue (Table 27) was calculated from the concentration of each lanthanide in the residual solution and in the feed. On the average, about 0.14% of the feed remained in the feed tank after displacement by 60 liters of water; this compares favorably with an expected residue of <0.5% based on preliminary tests not reported here.

Two additional feed displacement tests, Runs 3 and 4,* were carried out with solutions containing neodymium nitrate and nitric acid that had been evaporated, steam-stripped, and denitrated with formic acid during previous evaporator chemical tests (Runs 4 and 5, Table 17). For the Run 3 displacement test, feed solution from evaporator Run 4 was displaced from the column feed tank with 40 liters of water. For the Run 4 displacement test, feed solution from evaporator Run 5 was displaced with 20 liters of water. After water displacement, the residue in the column feed tank was mixed and the neodymium concentration determined. Based on the neodymium analyses, 0.008% to 0.01% of the Run 3 feed remained in the column feed tank; this residue appears to be low by a factor of 10 or more when compared with residues from Runs 1, 2, and 4, and is significantly less than the 2% estimated from preliminary 40-liter displacement tests not reported here. For Run 4, neodymium analyses indicated that displacement by 20 liters of water left 0.18% to 0.24% of the feed in the column feed tank.

The Dowex® 50W-X8 cation-exchange resin in the columns is compacted about 5% by the high pressures required for flow through the resin beds. The resin volume also shrinks about 10% when converted from the H^+ form (saturated with H^+) to the Zn^{2+} form (saturated with Zn^{2+}) and expands about 10% when converted from the Zn^{2+} form back to the H^+ form. These variations in resin volume prevent complete filling of the column cavities between the retainer plates with resin. The columns were filled with resin in the H^+ form and then conditioned with 0.5M $Zn(NO_3)_2$ solution to convert the resin to the Zn^{2+} form. Then the resins were compacted by the high pressure required for flow through the beds. The volumes of the compacted Zn^{2+} -form resin beds in the columns were measured by x-ray examination and compared with the measured volumes of resin loaded into and removed from the columns (Table 28).

* Elution from the resin columns was not tested.

TABLE 27

Percent of Feed Left in Feed Tank After
Water Displacement to RIX-I Columns

Lanthanide	Residue, % of Feed ^a	
	Run 1	Run 2
Dysprosium	0.15	0.23
Terbium	0.08	0.09
Gadolinium	0.13	0.18
Europium	0.20	0.12
Samarium	0.13	0.09
Neodymium	0.11 to 0.15	0.11 to 0.14
Average	0.14	0.14

a. Calculated from concentrations of lanthanides in feed tank before and after water displacement of feed solution to columns.

TABLE 28

Volumes of Dowex® 50W-X8 Resin in RIX-I Columns

Column	Calculated Volume of Column Cavity, L	Resin Volume, L			
		H ⁺ Form Loaded Into Columns	Compacted Zn ²⁺ Form in Columns ^a	Zn ²⁺ Form Removed From Columns ^b	After Reconversion From Zn ²⁺ to H ⁺ Form
1"	0.675	0.675	0.595	0.625	0.680
2"	2.31	2.68	2.11	2.28	2.44
3"	5.11	-	4.06	4.06	4.52
4"	8.90	8.08	7.07	7.33	8.17

a. By x-ray examination of loaded columns after pressurization.

b. No longer under pressure.

SYSTEM FLUSH

Before Frame 4 was returned to Construction, all process vessels and all process lines that had been contacted with any solution other than deionized water were thoroughly flushed with deionized water. All cooling water lines and heating water lines were flushed with nitric acid and then were flushed repeatedly with water.

APPENDIX A

Equipment List

Table A-1 lists the equipment piece numbers and the drawing numbers for the principal processing equipment in Frame 4.

TABLE A-1

Equipment List

<u>Equipment Piece (EP) Number</u>	<u>Name</u>	<u>Nominal Capacity, L</u>	<u>Process and Instruments Diagram Numbers</u>	<u>Detail Drawing</u>
10-1-5	Evaporator Transfer Tank	80	W-238731	D-146531
10-1-8E	RIX-I Feed Evaporator	125	W-238731	D-146775
10-1-9	Condenser	—	W-238731	D-146772
10-1-10	Condensate Tank	80	W-238731	D-146533
10-3-1	RIX-I Feed Run Tank	20	W-238732	D-146534
10-3-1-1	Rupture Disc	—	W-238732	—
10-3-2	Delay Pot	6	W-238732	D-146716
10-3-3	RIX-I Dump Tank	20	W-238732	D-146532
10-3-10	4" Column	9.9	W-238732	D-146726
10-3-11	3" Column	5.5	W-238732	D-146727
10-3-12	2" Column	2.5	W-238732	D-146728
10-3-13	1" Column	0.6	W-238732	D-146729
10-3-14	Rupture Disc	—	W-238732	—
10-3-20	RIX-I Waste Tank	500	W-238732	D-146559
10-9-1	Cf RIX-I Run Tank	75	W-238735	D-146722
10-100-1	AVR-093 Air Jet	—	W-238732	BPF-211915
10-100-5	Filter	—	W-238731	—
10-100-8	Rupture Disc	—	W-238732	—

APPENDIX B

Water Transfer Tests

Table B-1 lists measured water volumes and flow rates during hydraulic tests of various transfer paths between Frame 4 vessels.

TABLE B-1

Water Transfer Tests of Frame 4 Vessels

Transfer Path ^a	Vacuum, inches of mercury	Water Transfer		
		Volume, L	Time, min	Rate, L/min
Feed Adjustment Evaporator to Feed Tank	26	17.9	5.6	3.2
Feed Tank to Evaporator Transfer Tank	25	17.9	10.0	1.8
Evaporator Transfer Tank to Feed Adjustment Evaporator	—	51.0	33.5	1.5
Condensate Tank to Evaporator Transfer Tank	20	41.0	10.8	3.8
Cf Raw Feed Evaporator ^b to Evaporator Transfer Tank	20	50.0	16.6	3.0
Waste Tank to Waste Header	26	97.5	49.0	2.0
	22	270.0	127.0	2.1
Condensate Tank to Waste Header	20	48.1	26.9	1.8
Cf Run Tank to Cf Transfer Tank ^c	20	30.2	15.5	2.0
Dump Tank to Rerun Transfer Tank ^d	18	15.0	10.0	1.5
Cold Feed Drops to Sump Flush	— ^e	10.3	4.2	2.5

a. See Figure 1.

b. EP 17.3 in the canyon. Simulated.

c. EP 10-9-3 on Frame 5. Simulated.

d. EP 10-23-1 on Frame 5. Simulated.

e. 15 psig pressure to simulate gravity drop.

APPENDIX C

Measurement of Vessel Off-Gas Exhaust Flows

Each vessel was isolated and the off-gas flow was determined by measuring the pressure drop across an orifice of appropriate size temporarily installed in the off-gas header. Pressure drops were measured at various static vacuums maintained in the header while internally generated air flowed into the vessel at various rates. Off-gas flows were calculated from these measurements by the following formula:³

$$W = KYA \sqrt{2g_c(P_1 - P_2)}d$$

Where W = mass flow, lb/sec

$$K = C/\sqrt{1 - R^4}, \text{ dimensionless}$$

C = dimensionless coefficient of discharge

= 0.61 for this study

R = diameter of orifice/diameter of pipe

$$Y = 1 - \left[\left(\frac{1 - P_2/P_1}{C_p/C_v} \right) \left(0.41 - 0.35R^4 \right) \right]$$

C_p/C_v = specific heat ratio

= 1.40 for air

P_1 = absolute upstream pressure, (lb force)/ft²

P_2 = absolute downstream pressure, (lb force)/ft²

A = cross-sectional area of orifice, ft²

g_c = gravitational acceleration

= 32.17 (lb mass)(ft)/(lb force)(sec²)

d = density of upstream air, (lb mass)/ft³

3. J. H. Perry, Ed. *Chemical Engineers' Handbook*. Fourth edition, Section 5-10, McGraw-Hill, New York (1963).

Results are listed in Table C-1. Air flows into vessel overflow lines were spot-checked (results not listed) with a Type 3002 Velometer® (Alnor Instrument Company).

TABLE C-1

Off-Gas Exhaust Flows From Frame 4 Tanks

Tank EP Number	Air Sparge to Vessel, scfm	Measured Off-Gas Flow, scfm, at Header Vacuum, inches of water, of					Design Flow, ^a scfm
		0.05	0.10	0.20	0.30	0.40	
10-3-20	0	1.8	2.6	3.7	4.6	5.3	3.75
	1.0	2.6	3.4	4.5	5.4	6.0	3.75
	2.4	3.2	3.8	5.3	6.0	6.8	3.75
10-3-3	0	1.0	1.5	1.8	2.5	2.7	1.25
	1.0	1.1	1.6	2.0	2.7	2.9	1.25
	2.2	--	--	2.2	2.8	3.2	1.25
10-1-10	0	1.5	2.2	2.9	3.8	4.5	1.67
	1.0	2.1	2.8	3.5	4.5	5.1	1.67
	2.4	2.55	3.2	4.1	5.1	5.6	1.67
10-9-1	0	1.5	2.2	3.2	3.8	4.4	1.67
	1.0	2.1	2.8	3.8	4.4	5.1	1.67
	2.3	2.5	3.4	4.4	5.1	4.6	1.67

a. Minimum flow required.

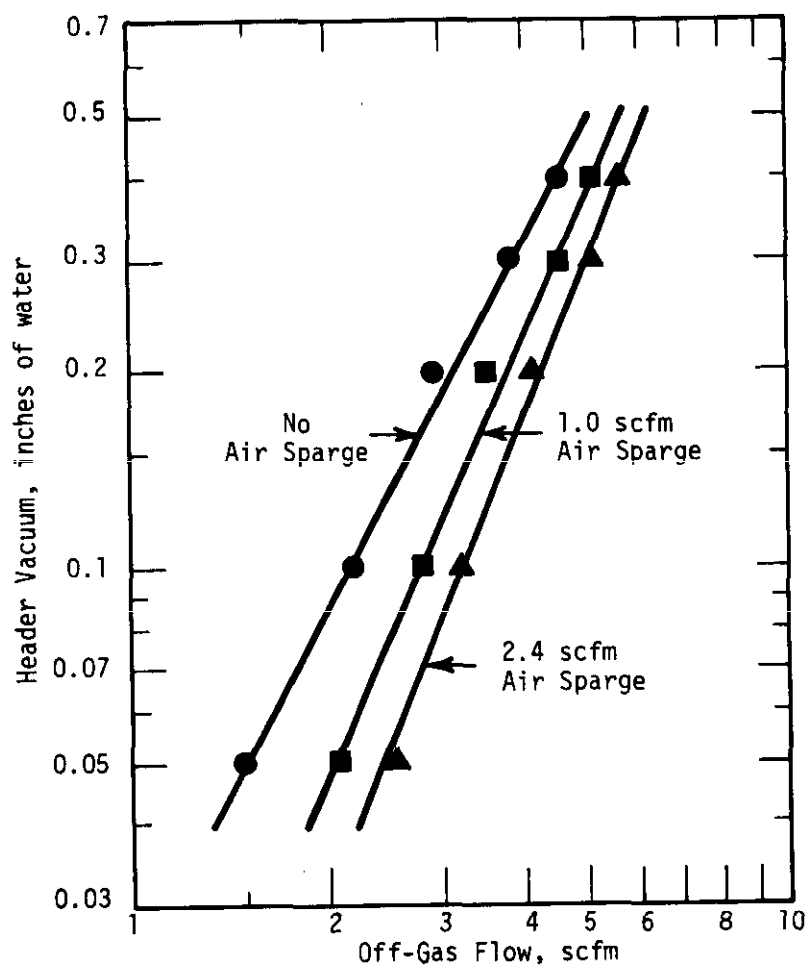


FIGURE C-1. Off-Gas Exhaust Flow from Condensate Tank (EP 10-1-10)

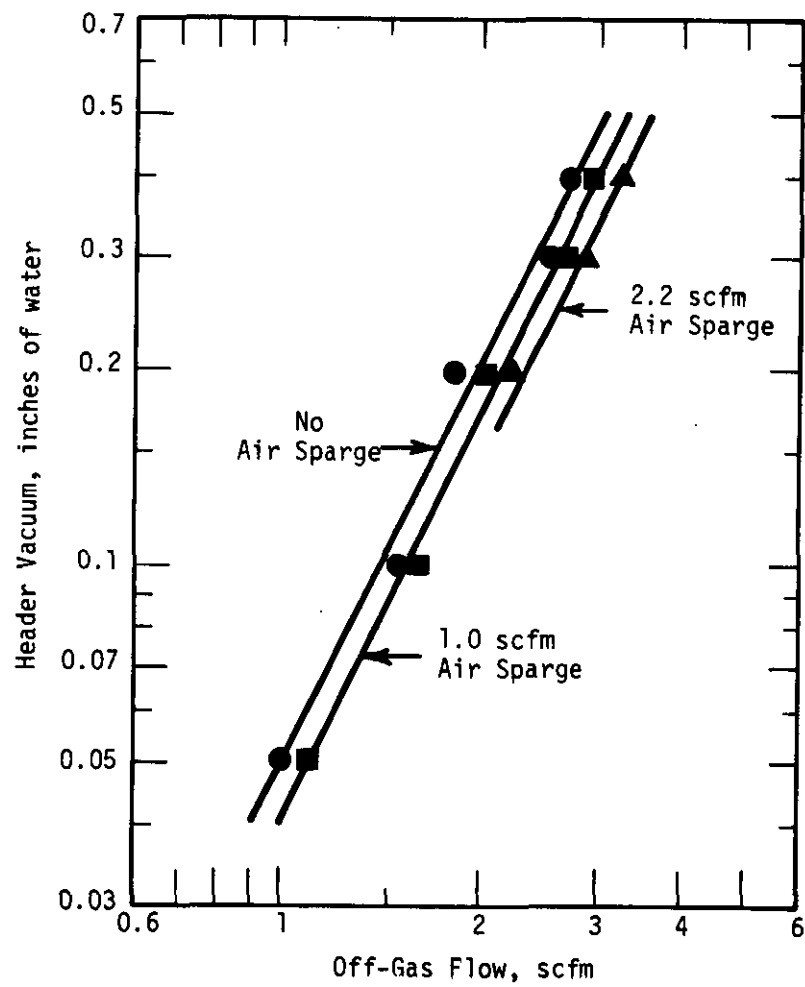


FIGURE C-2. Off-Gas Exhaust Flow from RIX-I Dump Tank (EP 10-3-3)

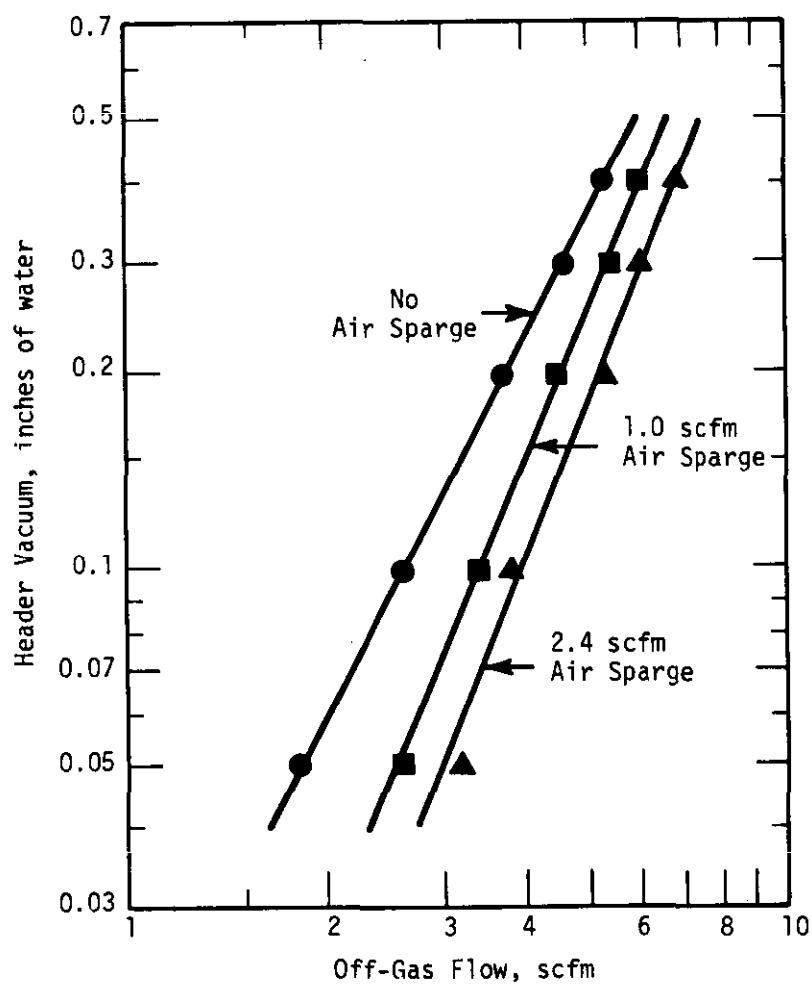


FIGURE C-3. Off-Gas Exhaust Flow from RIX-I Waste Tank (EP 10-3-20)

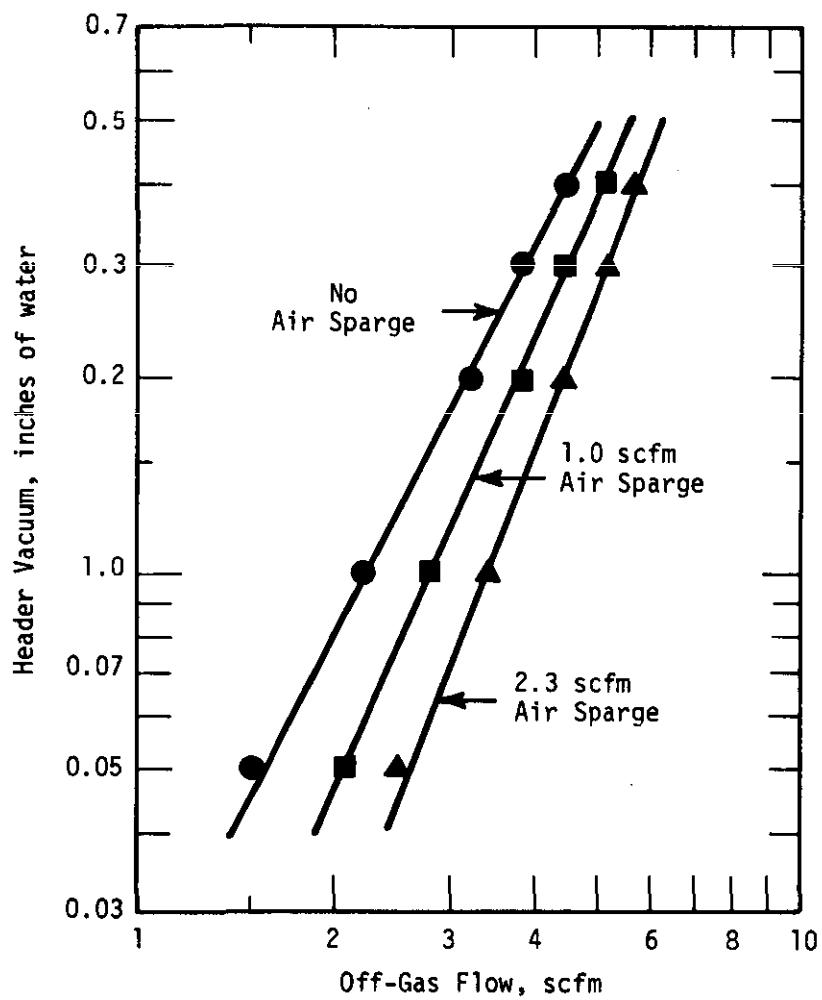


FIGURE C-4. Off-Gas Exhaust Flow from Cf RIX-I Run Tank (EP 10-9-1)

APPENDIX D

Evaporation, Steam Stripping, and Denitration

Tables D-1 through D-6 list the results of the cold chemical runs in the feed adjustment evaporator.

TABLE D-1

Run 1 Evaporation, Steam Stripping, and Denitration

	Makeup	After Evaporation	After Steam Stripping						After Denitration with 2.62 L of 23.4M HCOOH
			First	Second	Third	Fourth	Fifth	Sixth	
Bottoms in 10-1-8E Evaporator									
Volume, L	50	9.25	14.0	9.25	10.5	11.1	11.5	10.4	9.1
H ⁺ molarity in samples	2.7	11.5	5.8	7.9	5.9	4.7	4.3	4.69	2.32
	2.7	11.5	5.8	7.9	5.9	4.7	4.3	4.69	2.32
Average H ⁺ molarity	2.7	11.5	5.8	7.9	5.9	4.7	4.3	4.69	2.32
H ⁺ , moles	135	106.38	81.20	73.08	61.95	52.17	49.45	48.78	21.11 ^a
H ⁺ , % of initial	100	82.3	74.0	89.8	83.2	87.9	94.3	89.9	—
Overheads in Condensate Tank 10-1-10									
Volume, L	—	58.6	16.8	14.5	27.7	27.5	27.0	25.8 ^b	—
H ⁺ molarity in samples	—	0.39	1.7	0.57	0.46	0.26	0.11	0.21 ^b	—
	—	0.39	1.7	0.57	0.44	0.26	0.11	0.21 ^b	—
Average H ⁺ molarity	—	0.39	1.7	0.57	0.45	0.26	0.11	0.21 ^b	—
H ⁺ , moles	—	22.85	28.56	8.27	12.47	7.15	2.97	5.48 ^b	—
H ⁺ , % of initial	—	17.7	26.0	10.2	16.8	12.1	5.7	10.1	—
Material Balance									
Based on H ⁺ , %	—	95.7	103	100	102	95.8	100	110	—

a. $\frac{\text{Moles of Formic Acid}}{\text{Moles of H}^+ \text{ Decomposed}} = \frac{(2.62)(23.4)}{(48.78 - 21.11)} = 2.22$

- b. Condensate from the fifth stripping was not pumped out of Tank 10-1-10 before receipt of condensate from the sixth stripping. The volume and H⁺ content of condensate from the sixth stripping were therefore calculated from the total, volume of condensate from the fifth and sixth strippings (52.8 L) and H⁺ concentration measured in two samples (0.16M 0.16M) of this combined solution.

TABLE D-2

Run 2 Evaporation, Steam Stripping, and Denitration

	<u>Makeup</u>	<u>After Evaporation</u>	<u>After Steam Stripping</u>		<u>After Denitration with 2.55 L of 23.4M HCOOH</u>
			<u>First</u>	<u>Second</u>	
Bottoms in 10-1-8E Evaporator					
Volume, L	50	6.75	10.4	10.4	10.1
H ⁺ molarity in samples	2.64	13.02	6.12	4.61	1.85
	2.64	13.08	6.12	4.61	1.80
Average H ⁺ molarity	2.64	13.05	6.12	4.61	1.825
H ⁺ , moles	132.00	88.09	63.6	47.94	18.43 ^a
H ⁺ , % of initial	100	65.3	68.9	87.1	—
Overheads in Condensate Tank 10-1-10					
Volume, L	—	61.6	32.3	33.9	—
H ⁺ molarity in samples	—	0.76	0.896	0.21	—
	—	0.76	0.886	0.21	—
Average H ⁺ molarity	—	0.76	0.891	0.21	—
H ⁺ , moles	—	46.82	28.78	7.12	—
H ⁺ , % of initial	—	34.7	31.1	12.9	—
Material Balance					
Based on H ⁺ , %	—	102	105	86.6	—

a.
$$\frac{\text{Moles of Formic Acid}}{\text{Moles of H}^+ \text{ Decomposed}} = \frac{(2.55)(23.4)}{(47.94 - 18.43)} = 2.02$$

TABLE D-3

Run 3 Evaporation, Steam Stripping, and Denitration

	Makeup	After Evaporation	After Steam Stripping		After Denitration with 2.545 L of 23.4M HCOOH
			First	Second	
Bottoms in 10-1-8E Evaporator					
Volume, L	50	7.5	10.25	9.75	9.75
H ⁺ molarity in samples	2.69	12.40	5.27	4.85	2.15
	2.69	12.55	5.29	4.85	2.15
Average H ⁺ molarity	2.69	12.475	5.28	4.85	2.15
H ⁺ , moles	134.5	93.56	54.12	47.29	20.96 ^a
H ⁺ , % of initial	100	71.0	57.8	87.0	—
NO ₃ ⁻ molarity in samples	NA ^b	NA	4.77	4.28	0.50
	NA	NA	4.77	4.28	0.83
Average NO ₃ ⁻ molarity	—	—	4.77	4.28	0.665
NO ₃ ⁻ , moles	—	—	48.89	41.73	6.48 ^c
NO ₃ ⁻ , % of initial	—	—	62.0	96.0	—
Overheads in Condensate Tank 10-1-10					
Volume, L	—	61.6	31.6	35.2	—
H ⁺ molarity in samples	—	0.66	1.26	0.20	—
	—	0.58	1.24	0.20	—
Average H ⁺ molarity	—	0.62	1.25	0.20	—
H ⁺ , moles	—	38.19	39.5	7.04	—
H ⁺ , % of initial	—	29.0	42.2	13.0	—
NO ₃ ⁻ molarity in samples	—	NA	0.95	0.05	—
	—	NA	0.95	0.05	—
Average NO ₃ ⁻ molarity	—	—	0.95	0.05	—
NO ₃ ⁻ , moles	—	—	30.02	1.76	—
NO ₃ ⁻ , % of initial	—	—	38.0	4.0	—
Material Balance, %					
Based on H ⁺	—	97.6	100	100	—
Based on NO ₃ ⁻	—	—	84.3	89.0	—

a. $\frac{\text{Moles of Formic Acid}}{\text{Moles of H}^+ \text{ Decomposed}} = \frac{(2.545)(23.4)}{(47.27 - 20.96)} = 2.26$

b. NA = not analyzed.

c. $\frac{\text{Moles of Formic Acid}}{\text{Moles of NO}_3^- \text{ Decomposed}} = \frac{(2.545)(23.4)}{(41.73 - 6.48)} = 1.69$

TABLE D-4

Run 4A Evaporation, Steam Stripping, and Denitration

	After Evaporation ^a	After Steam Stripping ^b			Fourth	After Denitration with 1.07 L of 23.4M HCOOH
		First	Second	Third		
Bottoms in 10-1-8E Evaporator						
Volume, L	7.05	7.85	8.0	7.75	7.75	7.35
H ⁺ molarity in samples	12.34	5.17	4.35	3.82	3.64	2.18
	12.18	5.17	4.35	3.85	3.38	2.19
					3.40	2.20
					3.38	2.20
Average H ⁺ molarity	12.26	5.17	4.35	3.835	3.45	2.192
H ⁺ , moles	86.43	40.58	34.80	29.72	26.74	16.11 ^c
H ⁺ , % of initial	66.2	44.1	83.3	86.8	88.8	—
NO ₃ ⁻ molarity in samples	11.65	6.18	5.28	3.40	4.67	2.08
	11.96	6.05	5.35	NA ^d	4.54	2.08
					4.51	3.10
					3.95	3.10
Average NO ₃ ⁻ molarity	11.805	6.115	5.315	3.40	4.418	2.59
NO ₃ ⁻ , moles	83.23	48.00	42.52	26.35	34.19	19.03 ^e
NO ₃ ⁻ , % of initial	—	46.1	84.9	85.9	81.2	—
Nd ³⁺ molarity in samples	NA	0.225	0.24	0.24	0.22	0.25
	NA	0.225	0.24	0.23	0.21	0.25
					0.25	NA
					0.23	NA
Overheads in Condensate Tank 10-1-10						
Volume, L	59.7	59.3	34.2	34.6	33.6	—
H ⁺ molarity in samples	0.738	0.869	0.204	0.133	0.09	—
	0.743	0.869	0.205	0.129	0.11	—
Average H ⁺ molarity	0.7405	0.869	0.2045	0.131	0.10	—
H ⁺ , moles	44.21	51.53	6.99	4.53	3.36	—
H ⁺ , % of initial	33.8	55.9	16.7	13.2	11.2	—
NO ₃ ⁻ molarity in samples	NA	0.975	0.214	0.12	0.267	—
	NA	0.918	0.228	0.13	0.237	—
Average NO ₃ ⁻ molarity	—	0.9465	0.221	0.125	0.252	—
NO ₃ ⁻ , moles	—	56.13	7.56	4.33	7.96	—
NO ₃ ⁻ , % of initial	—	53.9	15.1	14.1	18.8	—
Nd ³⁺ molarity in samples	<7x10 ⁻⁶	ND ^f	<7x10 ⁻⁶	ND	2.8x10 ⁻⁵	—
	<7x10 ⁻⁶	ND	<7x10 ⁻⁶	<7x10 ⁻⁶	2.8x10 ⁻⁵	—

TABLE D-4 (Cont'd)

	After <u>Evaporation^a</u>	After Steam Stripping ^b				After Denitration with 1.07 L of <u>23.4M HCOOH</u>
		<u>First</u>	<u>Second</u>	<u>Third</u>	<u>Fourth</u>	
Material Balance, %						
Based on H^+	99.7	107	103	98.4	101	—
Based on NO_3^-	93.0	125	104	72.2	160	—

a. Original makeup data unavailable.

b. Water for steam stripping was added through spare dip leg #SP-204, which goes to the bottom of the evaporator, rather than as usual through the CFD seal pot #65, which is 5 inches shorter.

c. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } H^+ \text{ Decomposed}} = \frac{(1.07)(23.4)}{(26.74 - 16.11)} = 2.36$

d. NA = not analyzed.

e. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } NO_3^- \text{ Decomposed}} = \frac{(1.07)(23.4)}{(34.29 - 19.03)} = 1.64$

f. ND = not detected.

TABLE D-5

Run 4B Second Denitration

	After Evaporation, Steam Stripping, and First Denitration	After Second Denitration with 0.475 L of 23.4M HCOOH
Bottoms in 10-1-8E Evaporator		
Volume, L	7.08	9.08
H ⁺ molarity in samples	2.26 2.24 2.25 2.20	1.53 ^a 1.37 1.35 1.35
Average H ⁺ molarity	2.238	1.357
H ⁺ , moles	15.84	12.32 ^b
NO ₃ ⁻ molarity in samples	NA ^c NA 3.1 3.0	NA NA 2.0 1.96
Average NO ₃ ⁻ molarity	3.05	1.98
NO ₃ ⁻ , moles	21.59	17.98 ^d
Nd ³⁺ molarity in samples	0.26 0.26 NA NA	0.22 0.22 NA NA

a. Omitted from average.

$$b. \frac{\text{Moles of Formic Acid}}{\text{Moles of H}^+ \text{ Decomposed}} = \frac{(0.475) (23.4)}{(15.84 - 12.32)} = 3.16$$

c. NA = not analyzed.

$$d. \frac{\text{Moles of Formic Acid}}{\text{Moles of NO}_3^- \text{ Decomposed}} = \frac{(0.475) (23.4)}{(21.59 - 17.98)} = 3.08$$

TABLE D-6

Run 5 Evaporation, Steam Stripping, and Denitration

	Makeup	After Evaporation	After Steam Stripping				After Denitration with 1.75 L of 23.4M HOOH
			First	Second	Third	Fourth	
Bottoms in 10-1-8E Evaporator							
Volume, L	59.0	~9.4	7.85	8.35	8.15	8.25	9.5
H ⁺ molarity in samples	2.60	11.82	5.33	4.11	3.53	3.11	1.09
	2.94	11.03	5.33	4.11	3.57	3.13	1.09
	—	—	—	—	—	—	1.11
	—	—	—	—	—	—	1.11
Average H ⁺ molarity	2.77	11.425	5.33	4.11	3.55	3.12	1.10
H ⁺ , moles	163.43	107.40	41.84	34.32	28.93	25.74	10.45 ^a
H ⁺ , % of initial	100	70.8	48.8	79.2	87.0	90.9	—
NO ₃ ⁻ molarity in samples	NA ^b	9.68	6.11	4.95	4.48	4.26	1.60
	NA	12.58	6.22	5.06	4.50	4.24	1.82
	—	—	—	—	—	—	1.58
	—	—	—	—	—	—	1.58
Average NO ₃ ⁻ molarity	—	11.13	6.165	5.005	4.49	4.25	1.645
NO ₃ ⁻ , moles	—	104.62	48.40	41.79	36.59	35.06	15.63 ^c
NO ₃ ⁻ , % of initial	—	69.9	48.4	77.4	84.6	—	—
Nd ³⁺ molarity in samples	0.04	0.30	0.24	0.23	0.23	0.21	0.22
	0.04	0.30	0.23	0.22	0.23	0.25	0.23
	—	—	—	—	—	—	0.18
	—	—	—	—	—	—	0.18
Overheads in Condensate Tank 10-1-10							
Volume, L	—	52.0	33.0	34.0	37.5	34.2	—
H ⁺ molarity in samples	—	0.80	1.27	0.16	0.16	0.08	—
	—	0.90	1.39	0.37	0.07	0.07	—
Average H ⁺ molarity	—	0.85	1.33	0.265	0.115	0.075	—
H ⁺ , moles	—	44.20	43.89	9.01	4.31	2.57	—
H ⁺ , % of initial	—	29.2	51.2	20.8	13.0	9.1	—
NO ₃ ⁻ molarity in samples	—	0.869	1.39	0.413	0.206	NA	—
	—	0.862	1.74	0.306	0.150	NA	—
Average NO ₃ ⁻ molarity	—	0.8655	1.565	0.3595	0.178	—	—
NO ₃ ⁻ , moles	—	45.01	51.65	12.22	6.68	—	—
NO ₃ ⁻ , % of initial	—	30.1	51.6	22.6	15.4	—	—
Nd ³⁺ molarity in samples	—	ND ^d	<7x10 ⁻⁶	<7x10 ⁻⁶	<7x10 ⁻⁶	9x10 ⁻⁵	—
	—	ND	<7x10 ⁻⁶	4.2x10 ⁻⁵	ND	3x10 ⁻⁵	—

TABLE D-6 (Cont'd)

Material Balance, %	Makeup	After Evaporation	After Steam Stripping				After Denitration with 1.75 L of 23.4M HCOOH
			First	Second	Third	Fourth	
Based on H^+	—	92.8	79.8	104	96.9	97.8	
Based on NO_3^-	—	—	95.6	112	104	—	

a. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } H^+ \text{ Decomposed}} = \frac{(1.75)(23.4)}{(25.74 - 10.45)} = 2.68$

b. NA = not analyzed.

c. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } NO_3^- \text{ Decomposed}} = \frac{(1.75)(23.4)}{(35.06 - 15.63)} = 2.11$

d. ND = not detected.

TABLE D-7

Run 6 Denitration Without Evaporation or Steam Stripping

	Makeup	After Flushing into Evaporator with two 1.5-L Portions of H_2O	After Denitration with 1.925 L of 23.4M HCOOH
Solution Volume, L	7.0	9.88	10.25
H^+ molarity in samples	—	3.90	1.93
	—	3.90	1.93
	—	—	1.82
	—	—	1.84
Average H^+ molarity	5.81 ^a	3.90	1.875
H^+ , moles	40.67 ^b	38.53	19.27 ^c
NO_3^- molarity in samples	—	4.61	2.48
	—	4.64	2.44
	—	—	2.29
	—	—	2.37
Average NO_3^- molarity	6.48 ^a	4.625	2.395
NO_3^- , moles	45.36 ^b	45.70	24.55 ^b
Nd^{3+} molarity in samples	0.28 ^b	0.20	0.17
	—	0.20	0.17
	—	—	0.20
	—	—	0.17

a. Average of two samples, individual analyses not shown.

b. Prepared to contain 40.00 moles of H^+ , 46 moles of NO_3^- , and 2 moles of Nd^{3+} in 7 liters.

c. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } H^+ \text{ Decomposed}} = \frac{(1.925)(23.4)}{(38.53 - 19.27)} = 2.34$

d. $\frac{\text{Moles of Formic Acid}}{\text{Moles of } NO_3^- \text{ Decomposed}} = \frac{(1.925)(23.4)}{(45.70 - 24.55)} = 2.13$

APPENDIX E

Feed Compositions for RIX-I Column Chemical Runs

Table E-1 shows the quantities of reagents for each column run which were dissolved in 6 liters of deionized water, diluted to a final volume of 10 liters with deionized water, and added to the RIX-I Feed Tank (EP 10-3-1). After mixing and sampling for analysis (Table E-2), each solution was displaced by deionized water into the four-inch column.

TABLE E-1

Feed Make-Up for RIX-I Column Runs 1 and 2

Lanthanides to Simulate Actinides	For Run 1		For Run 2	
	grams ^a	moles ^a	grams ^a	moles ^a
Dysprosium Nitrate [Dy(NO ₃) ₃ ·5H ₂ O]	351	0.800	219	0.499
Terbium Nitrate [Tb(NO ₃) ₃ ·6H ₂ O]	362	0.799	226	0.499
Gadolinium Nitrate [Gd(NO ₃) ₃ ·6H ₂ O]	271	0.600	169	0.374
Europium Nitrate [Eu(NO ₃) ₃ ·6H ₂ O]	89	0.200	56	0.126
Samarium Nitrate [Sm(NO ₃) ₃ ·6H ₂ O]	356	0.800	223	0.502
Neodymium Nitrate [Nd(NO ₃) ₃ ·6H ₂ O]	<u>351</u>	<u>0.801</u>	<u>219</u>	<u>0.500</u>
Total	1780	4.000	1112	2.500

a. Per 10 liters.

TABLE E-2

Analysis of Feed Solutions for RIX-I Column Runs 1 and 2

<u>Lanthanide</u>	<u>Concentration of Rare Earth, grams/liter</u>			
	<u>For Run 1</u>		<u>For Run 2</u>	
	<u>By Analysis</u>	<u>By Make-Up</u>	<u>By Analysis</u>	<u>By Make-Up</u>
Dysprosium	13.25	13.01	8.75	8.11
Terbium	9.40	12.69	0.65	7.92
Gadolinium	8.50	9.44	4.25	5.89
Europium	3.18	3.03	1.62	1.91
Samarium	10.76	12.04	7.90	7.54
Neodymium	<u>10.50</u>	<u>11.55</u>	<u>7.50</u>	<u>7.21</u>
Total	55.53	61.76	30.67	38.58

APPENDIX F

Displacement Chromatography Run 1 and 2

Tables F-1 and F-2 list the results of the two displacement chromatography tests of the RIX-I columns. Nonradioactive lanthanides were used to simulate the separation of actinides and fission-product lanthanides by the columns.

TABLE F-1

Analysis of Effluent from RIX-I Columns During Run 1

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
1	NA	NA	NA	NA	NA	NA
2	NA	NA	NA	NA	NA	NA
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ND	NA	NA	NA	NA	NA
6	ND	NA	NA	NA	NA	NA
7	ND	NA	NA	NA	NA	NA
8	0.10	NA	NA	NA	NA	NA
9	10.80	NA	NA	NA	NA	NA
10	8.70	NA	NA	NA	NA	NA
11	9.50	NA	NA	NA	NA	NA
12	10.30	NA	NA	NA	NA	NA
13	6.60	NA	NA	NA	NA	NA
14	6.80	ND	NA	NA	NA	NA
15	9.00	0.15	NA	NA	NA	NA

TABLE F-1 (Cont'd)

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
16	6.00	0.30	NA	NA	NA	NA
17	5.55	0.63	NA	NA	NA	NA
18	5.00	2.80	NA	NA	NA	NA
19	4.35	2.90	NA	NA	NA	NA
20	3.30	4.50	NA	NA	NA	NA
21	3.00	3.80	NA	NA	NA	NA
22	2.85	4.20	NA	NA	NA	NA
23	2.00	4.60	ND	ND	NA	NA
24	1.00	6.10	ND	ND	NA	NA
25	0.40	5.80	ND	ND	NA	NA
26	0.16	6.00	ND	ND	NA	NA
27	0.05	6.60	ND	ND	NA	NA
28	0.04	5.80	ND	ND	NA	NA
29	ND	8.30	ND	ND	NA	NA
30	ND	6.20	ND	ND	NA	NA
31	ND	6.40	ND	ND	ND	NA
32	ND	6.10	ND	ND	ND	NA
33	ND	4.15	0.43	0.65	ND	NA
34	ND	0.30	0.77	3.20	ND	NA
35	ND	ND	1.50	2.80	ND	NA
36	ND	ND	1.56	2.75	ND	NA
37	ND	ND	1.50	2.90	ND	NA

TABLE F-1 (Cont'd)

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
38	ND	ND	1.50	2.80	ND	NA
39	ND	ND	1.54	2.60	ND	NA
40	NA	ND	1.50	3.40	ND	NA
41	NA	ND	1.54	3.15	ND	NA
42	NA	ND	1.40	3.00	ND	NA
43	NA	ND	1.50	2.60	ND	NA
44	NA	ND	1.54	2.70	ND	NA
45	NA	ND	1.52	2.80	ND	NA
46	NA	ND	1.00	2.10	5.80	NA
47	NA	ND	0.009	ND	5.80	NA
48	NA	ND	ND	ND	6.50	NA
49	NA	ND	ND	ND	7.40	NA
50	NA	ND	ND	ND	6.00	NA
51	NA	ND	ND	ND	5.00	NA
52	NA	ND	ND	ND	5.80	NA
53	NA	ND	ND	ND	5.80	NA
54	NA	ND	ND	ND	5.60	NA
55	NA	ND	ND	ND	5.40	NA
56	NA	ND	ND	ND	5.40	ND
57	NA	ND	ND	ND	5.00	ND
58	NA	ND	ND	ND	5.00	ND

TABLE F-1 (Cont'd)

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
59	NA	ND	ND	ND	5.00	ND
60	NA	ND	ND	ND	2.80	1.20
61	NA	ND	ND	ND	ND	11.00
62	NA	ND	ND	ND	ND	4.90
63	NA	ND	ND	ND	ND	5.00
64	NA	ND	ND	ND	ND	4.90
65	NA	ND	ND	ND	ND	6.60
66	NA	NA	NA	NA	NA	5.30
67	NA	NA	NA	NA	NA	5.90
68	NA	NA	NA	NA	NA	6.10
69	NA	NA	NA	NA	NA	5.90
70	NA	NA	NA	NA	NA	6.40
71	NA	NA	NA	NA	NA	5.30
72	NA	NA	NA	NA	NA	5.50
73	NA	NA	NA	NA	NA	5.00
74	NA	NA	NA	NA	NA	5.00
75	NA	NA	NA	NA	NA	7.80
76	NA	NA	NA	NA	NA	2.00
77	NA	NA	NA	NA	NA	0.48
78	NA	NA	NA	NA	NA	ND

a. NA = sample not analyzed for this lanthanide.
 ND = lanthanide not detected in this sample.

TABLE F-2

Analysis of Effluent from RIX-1 Columns During Run 2

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
1	NA	NA	NA	NA	NA	NA
2	NA	NA	NA	NA	NA	NA
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ND	NA	NA	NA	NA	NA
7	ND	NA	NA	NA	NA	NA
8	ND	NA	NA	NA	NA	NA
9	ND	NA	NA	NA	NA	NA
10	2.86	NA	NA	NA	NA	NA
11	5.24	NA	NA	NA	NA	NA
12	5.24	NA	NA	NA	NA	NA
13	5.28	NA	NA	NA	NA	NA
14	5.28	ND	NA	NA	NA	NA
15	4.92	0.13	NA	NA	NA	NA
16	4.92	0.25	NA	NA	NA	NA
17	4.52	0.54	NA	NA	NA	NA
18	2.84	2.40	NA	NA	NA	NA
19	2.20	5.95	NA	NA	NA	NA
20	0.51	5.96	NA	NA	NA	NA
21	0.022	5.90	ND	ND	NA	NA
22	0.019	5.40	ND	ND	NA	NA
23	ND	5.40	ND	ND	NA	NA
24	ND	5.40	ND	ND	NA	NA
25	ND	6.20	ND	ND	NA	NA
26	ND	6.20	ND	ND	NA	NA
27	NA	6.15	ND	ND	NA	NA

TABLE F-2 (Cont'd)

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
28	NA	5.40	0.0385	ND	NA	NA
29	NA	0.70	1.10	3.96	NA	NA
30	NA	ND	1.29	4.065	NA	NA
31	NA	ND	1.33	3.98	NA	NA
32	NA	ND	1.36	3.25	NA	NA
33	NA	ND	1.36	3.79	NA	NA
34	NA	ND	1.36	3.13	ND	NA
35	NA	ND	1.38	4.27	ND	NA
36	NA	ND	1.356	3.90	ND	NA
37	NA	ND	1.356	3.07	ND	NA
38	NA	ND	1.388	3.06	ND	NA
39	NA	ND	1.416	3.715	ND	NA
40	NA	ND	0.364	ND	3.80	NA
41	NA	ND	ND	ND	4.015	NA
42	NA	ND	ND	ND	5.05	NA
43	NA	ND	ND	ND	4.90	NA
44	NA	ND	ND	ND	5.00	NA
45	NA	ND	ND	ND	5.00	NA
46	NA	ND	ND	ND	5.00	ND
47	NA	NA	NA	NA	5.005	ND
48	NA	NA	NA	NA	5.015	ND
49	NA	NA	NA	NA	4.82	ND
50	NA	NA	NA	NA	4.75	0.232
51	NA	NA	NA	NA	0.28	3.25
52	NA	NA	NA	NA	ND	3.35
53	NA	NA	NA	NA	ND	4.75

TABLE F-2 (Cont'd)

Volume of Accumulated Effluent from One-Inch Column, L	Concentration, grams/liter, in One-Liter Composite Samples ^a					
	Dy	Tb	Eu	Gd	Sm	Nd
54	NA	NA	NA	NA	ND	4.75
55	NA	NA	NA	NA	ND	4.60
56	NA	NA	NA	NA	NA	4.15
57	NA	NA	NA	NA	NA	4.30
58	NA	NA	NA	NA	NA	3.50
59	NA	NA	NA	NA	NA	4.35
60	NA	NA	NA	NA	NA	4.35
61	NA	NA	NA	NA	NA	2.58
62	NA	NA	NA	NA	NA	1.17
63	NA	NA	NA	NA	NA	0.36

a. NA = sample not analyzed for this lanthanide.
ND = lanthanide not detected in this sample.

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